**UNITED STATES ENVIRONMENTAL PROTECTION AGENCY**
**REGION V**

**DATE:** November 21, 1986

**SUBJECT:** Review of Quality Assurance Project Plan (QAPP) for the Reilly Tar and Chemical Corporation, N.P.L. Site, St. Louis Park, Minnesota

**FROM:** James H. Adams, Jr., Chief
Quality Assurance Office

**TO:** Norman Niedergang, Chief
CERCLA Enforcement Section

Attention: Dan Bicknell, RPM

Our Office has reviewed the Quality Assurance Project Plan (QAPP) for the Reilly Tar and Chemical Corporation N.P.L. Site, St. Louis Park, Minnesota, which our office received on October 14, 1986 (QAO #205). This QAPP is not acceptable because it contains several major deficiencies, which needs to be addressed. The goal and the scope of work for the project has not been clearly defined; and some of the required information are scattered throughout the QAPP, which makes the review of this QAPP very laborious. We suggest that this QAPP should be rewritten to incorporate all of the required information into each appropriate QAPP element per the EPA Guidance for QAPP preparation (QAMS-005/80).

The Quality Assurance Office will approve this subject QAPP when it is rewritten, and the following deficiencies are addressed.

I. Project Description

This QAPP element is not addressed. The project description should be addressed to include 1) project background including past data if any; 2) project objectives; 3) the intended data usage; 4) sampling rationale and design; 5) sample matrix and parameters to be tested, etc.

#9

II. Project Organization and Responsibility

For this QAPP element, we provide the following comments.

1. The organization chart (Figure 1-1) is not adequate. This chart should be expanded to include EPA, Region V as part of the organization.

2. The description of the project responsibilities is not sufficient. It should include the responsibilities for sample collection, field quality control, overall QC oversight, data assessment, etc. Identify them.

III. Quality Assurance Objectives

The QA Objectives is an individual QAPP element, in which the level of QA efforts required for the project is addressed. The description of Section 1.1 (Quality Objectives, page 2 of 55) is insufficient. The QA Objectives should include audits such as method blank, field blank, field duplicate, surrogate spike, matrix spike and matrix spike duplicate, etc. The data quality for each measurement system should be assessed in terms of accuracy, precision, completeness, etc. The QA Objectives should be site specific; and the acceptance limits for accuracy, precision and completeness, etc., should also be specified.

IV. Sampling Plan

The description of the sampling is insufficient. The sampling plan should be revised to include the followings:

1. Sample numbering system

   An adequate sample numbering system, which is required for the purpose of Chain-of-Custody, should be described.

2. The sampling points and actual number of samples to be collected shall be specified. Insertion of a table of sample summary is appropriate. Table 2-1 should be expanded to include all the parameters to be tested and the actual number of samples to be taken at each sampling location including field blanks and field duplicate.

3. Sampling procedures should be addressed for different samples to be collected. Sample filtration and preservation to be done in the field shall be clearly specified.

4. Field measurements
   Any field measurements to be performed during the sampling should be cleary addressed. The description of field measurements shall include at least the followings:

   a) Parameters to be monitored.

   b) Instruments to be used for each measurement.

c) Procedures including calibration and frequency.

d) Quality Control criteria.

V. Analytical procedure

The description of this QAPP element is insufficient. Our comments are as follows:

1. The chain-of-custody for laboratory activity is not addressed.

2. Not all of the analytical methodologies to be used are documented. Documentation of all analytical test procedures is necessary. EPA methods to be used can be specified by reference provided that the procedures are to be followed exactly. Any modification of the procedures should be documented. If non-EPA methods are to be used, they should be attached to the QAPP.

3. The sample preparation is not adequately addressed. The description of sample preparation should address all sample for different analyses. For example, it is stated that samples are to be analyzed for phenols; however, the sample preparation for this analysis is not described.

   In the sample preparation for ultratrace PAH analysis, it is stated that 4 1-liter samples will be divided into 2 2-liter portion. Each portion will be extracted with a 2-liter separatory funnel. We do not think this is a good procedure. It is our understanding that 2 liter of sample plus solvent (80 ml) will fill up the whole separatory funnel, which will leave very little space for the mixing of solvent with the sample body. As a result, the extract will not be complete and reproducible. We suggest that each one liter sample should be extracted separatedly using a 2-liter separatory funnel to ensure the completeness and reproducibility of the extraction. A total of 4 individual extraction will be required for 4 liter of sample.

4. Data Reduction, Validation and Reporting

   The followings should be addressed.

   1. It should be specified that any method used for data reduction should be documented, and should be part of the data reporting package.

   2. The criteria that will be used to validate data quality should be defined. It is stated (page 27 of 55) that ERT will validate the analytical data by utilizing the method spike sample criteria in conjunction with the surrogate

recovery criteria. This is not acceptable because the method spike recovery does not reflect the matrix effect, if any. The matrix spike recovery criteria should be used instead of method spike criteria. Correction should be made where it is appropriate.

3. The data reporting package to be used should also be specified.

VI. Calibration Procedures and frequency

The calibration procedures to be used and related information should be addressed under this QAPP element. It should includes not less than the followings:

1. The calibration procedure for each major measurement parameter should be provided either by reference to standard operational procedure (SOP), statement of work or by a written description of the calibration procedure.

2. The frequency of recalibration should be specified. This should include the conditions when a recalibration is required.

3. The calibration standrad solutions to be used should be addressed. This should includes the source from which the primary standard will be obtained, the components (or compounds) to be included in the solutions and their concentrations, and procedures for the preparation of working standards, etc.

VII. Internal Quality Control Checks

The description of the internal quality control checks should include no less than the followings:

1. Analysis of blanks (reagent blank, method blanks, field blanks, trip blank, etc.).

2. Use and analysis of internal standards.

   The compounds used for internal standard and their concentrations should be specified.

3. Instrument tuning and/or zeroing.

4. Analysis of quality control samples.

5. Surrogate samples

   Specify compounds used as surrogate and their concentration. The acceptance limits should also be specified.

6. Calibration standards - specify the composition and concentrations.

7. Calibration check standards - specify the composition and concentration. Define the acceptance limits as well.

8. Reagent Checks.

VIII. Chain-of-Custody

The final evidence file is not addressed. The Chain-of-Custody contians three major elements, mainly the field sampling, laboratory analysis and the final evidence file. All of three elements of Chain-of-Custody should be adequately addressed. This concern should be resolved by project manager.

IX. Performance and System Audits

The Performance and System Audits are not addressed in this QAPP. A section should be added to address both the Performance and System Audits. It should include the frequency of audits, Analysis Performance Evaluation (PE) sample, responsibilities for these audits and providing PE samples, etc. The description should be site-specific.

X. Preventive Maintenance

The preventive maintenance is not addressed. The description of the preventive maintenance should cover both laboratory equipment and field measurement equipments. Schedule of important preventive maintenance to be carried out to minimize the down time of the measurement system should be addressed. Such scheduled maintenance may include stock-up of critical spare parts, service contract with instrument manufacturer, etc.

XI. Corrective Action

The corrective action procedure must be addressed for each measurements. The description of corrective action should includes the followings:

1. The predetermined limits for data acceptability beyond which corrective action is required, should be specified.

2. Procedures with detailed steps to be taken should be described.

3. For each measurement system, identify the responsible individual for initiating the corrective action, and also the individual responsible for approving the corrective action.

XII. Data Assessment

The criteria or guideline to be used for data assessment should be specified. Identify the responsible individual for performing the data assessment.

XIII. Miscellaneous

1. The ERT SOP #7130 should be attached (page 14 of 55). ✓
2. We suggest that bottle blank should be analyzed for any contamination resulting from contaminated sample bottle (page 16 of 55).
3. Table 2-4 (page 17 of 55) should be revised to include only these appliable to the project.
4. Referencing document - Method for chemical analysis of water and wastes - is out-of-date. The current document is revised March, 1983 (page 46 of 55).
5. In Table 2-5 (page 20 of 55), the control limits are not specified.
6. In page 23 of 55, one bullet should be added for the matrix spike and matrix spike duplicate.
7. The modified EPA Method 625 should be attached.
8. In page 26 of 55, it should be specified that the surrogate spike and matrix spike should be added to the sample prior to the addition of solvent.
9. It is stated that only those surrogate recoveries which meet the acceptance criteria will be added to the control chart. This is not acceptable. All of the surrogate recoveries data should be added to the control chart.
10. Page 36 of 55, for matrix recovery sample, the actual sample should be used for spike instead of laboratory reagent water.
11. Section 4.1.6, the daily GC/MS performance tests should include the analysis of calibration check sample prior to the analysis of project sample, and should also be done at the beginning of every 12 hours shift.

12. Section 4 describes the expanded analysis to be taken; however, it is not clear *which* sample will be used for expanded analyses, what parameters to be tested, and when these expanded analyses will be performed. Needs clarification.

13. Table 4-7, the analytical methods to be used for $MH_3$, chloride and sulfate should be identified, for example, Method 350.1, 350.2 or 350.3.

14. The method detection limit & validation of the modified EPA Method 625 should be attached.

cc: M. Gade, WMD
T. Rutter, ERRB
S. Hong, CES

Meeting - 12/19/86

| | | |
|---|---|---|
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12-19-86

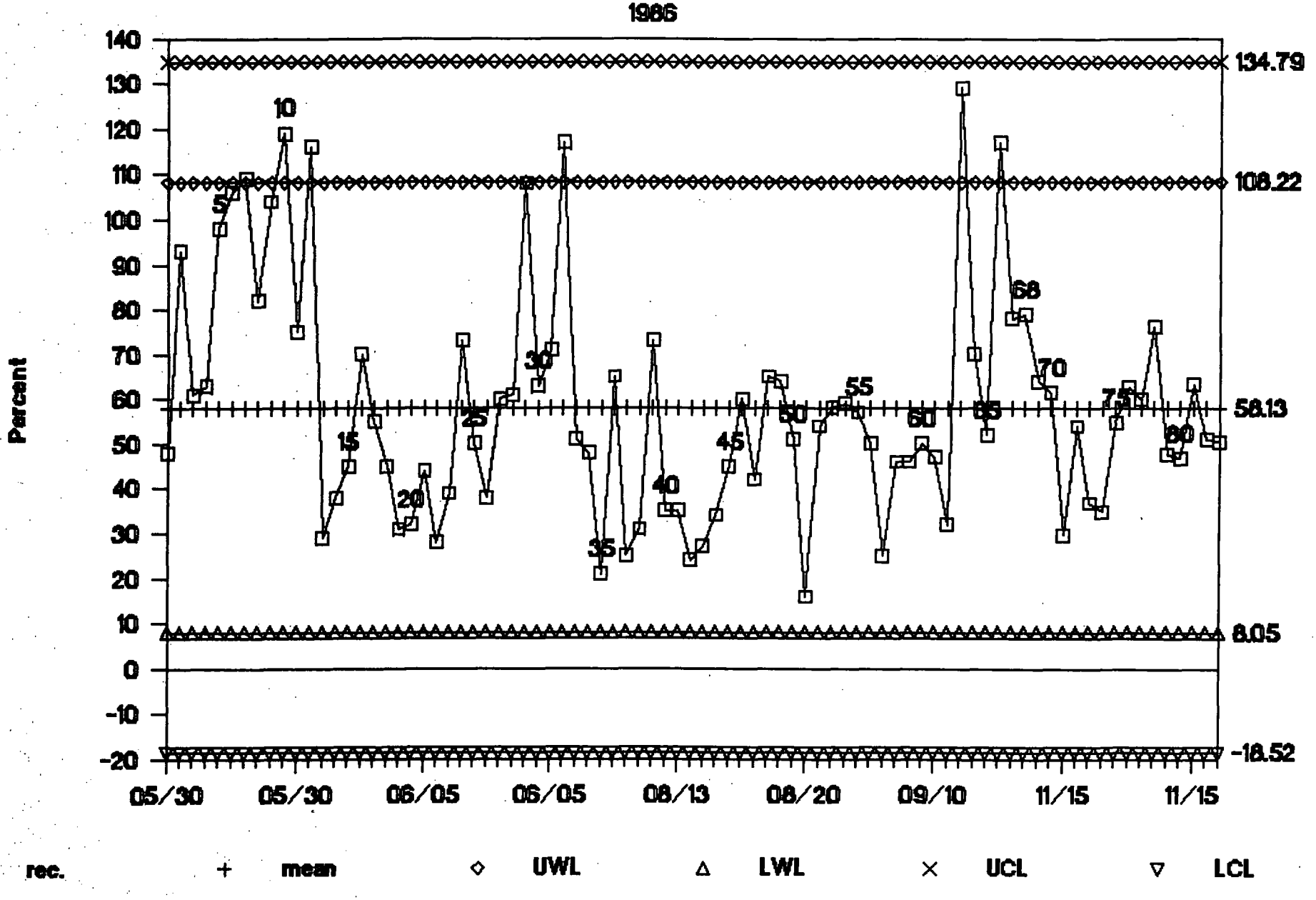
# SURROGATE LIMITS
# AS PRESENTED FOR THE CITY OF ST. LOUIS PARK, MN

| | July 1986 | October 1986 | All values |
|---|---|---|---|
| naphthalene-d8 | 42-102 | 14-305 | 8-108 |
| fluorene-d10 | 60-128 | 27-238 | 41-162 |
| chrysene-d12 | 10-54 | 13-512 | MDL-118 |

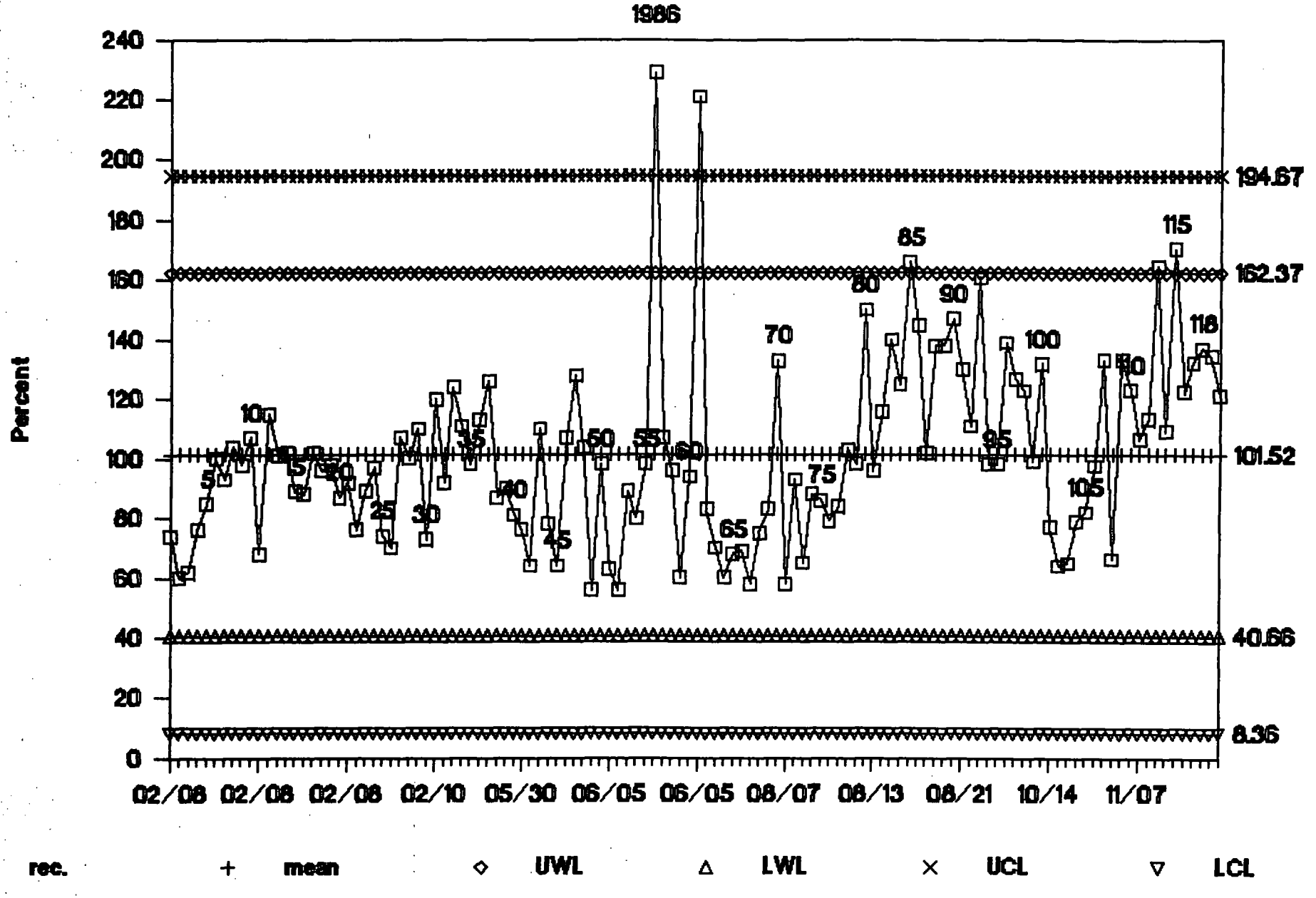
NOTE: these values are presented as the limits proposed in the July QAPP, the October QAPP (representing the limits prescribed in Method 1625 of the U.S. EPA Chemical Analysis Guidelines [40 CFR, Part 136; Federal Register, 26 October 1984, page 43425]), and the calculated values for the ERT surrogate recoveries from February through November 1986, respectively.

The July 1986 and "All values" ranges are the 95% confidence limits.

# Naphthalene Surrogate Control Chart

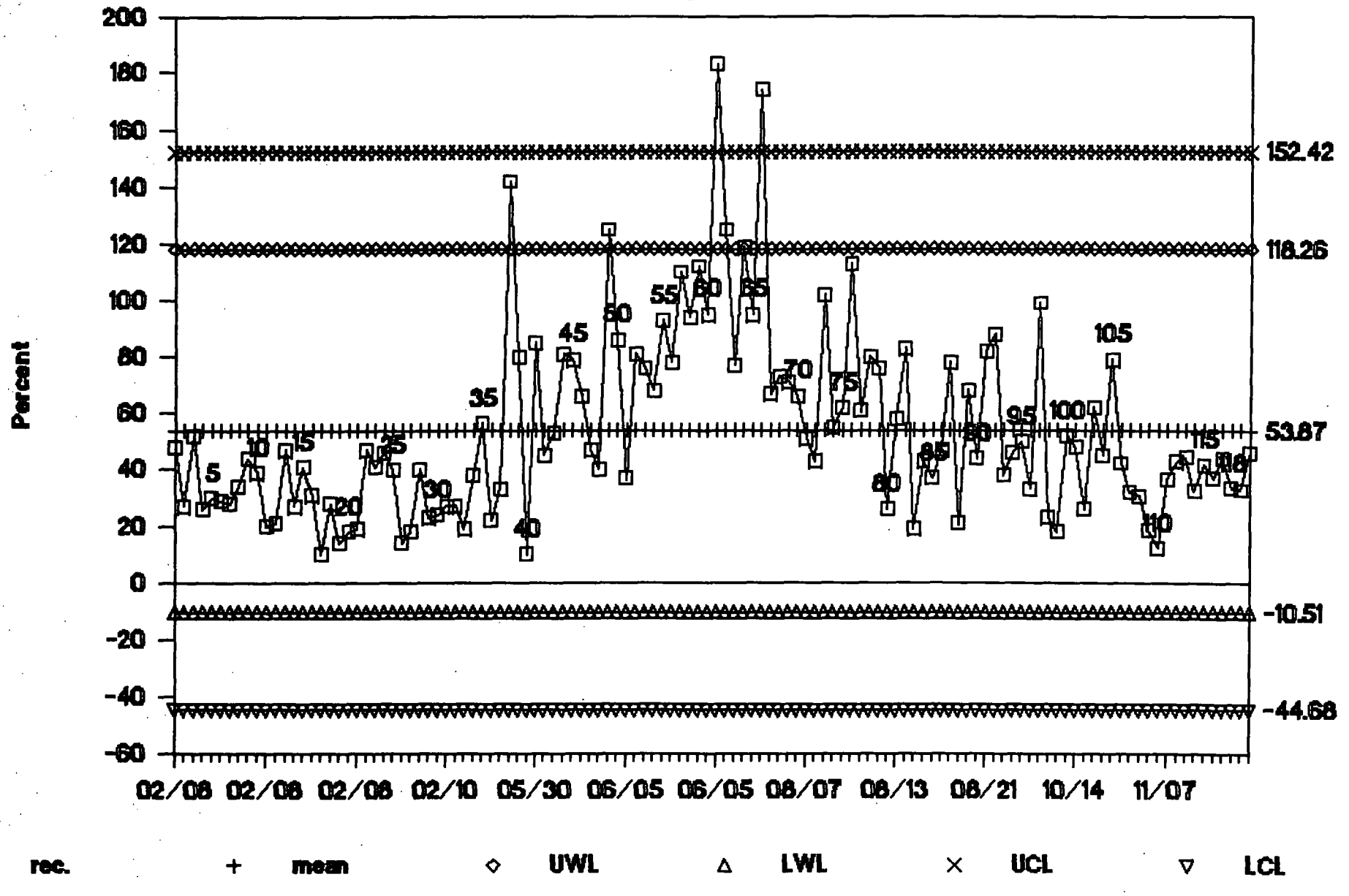


# Fluorene Surrogates Control Chart

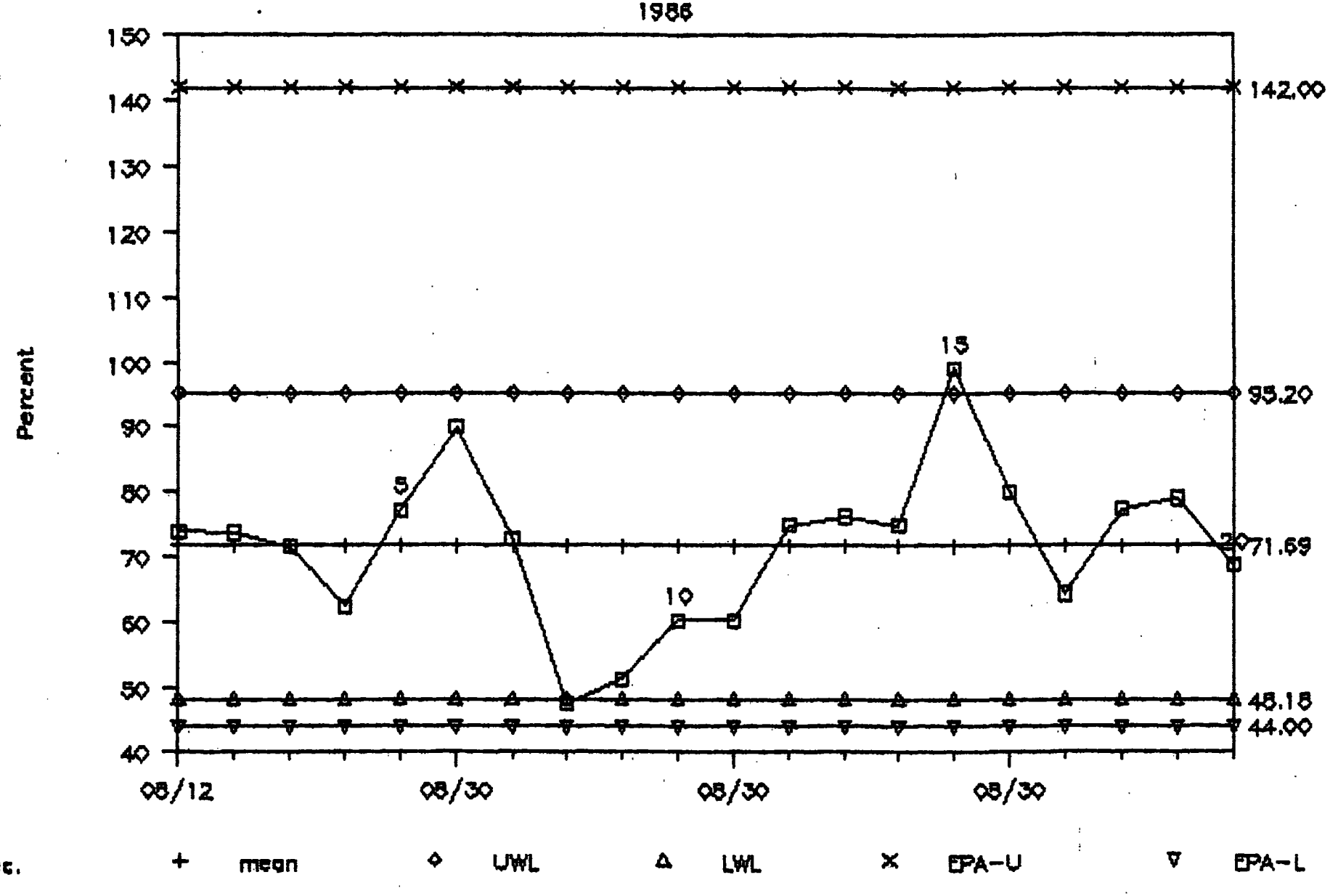


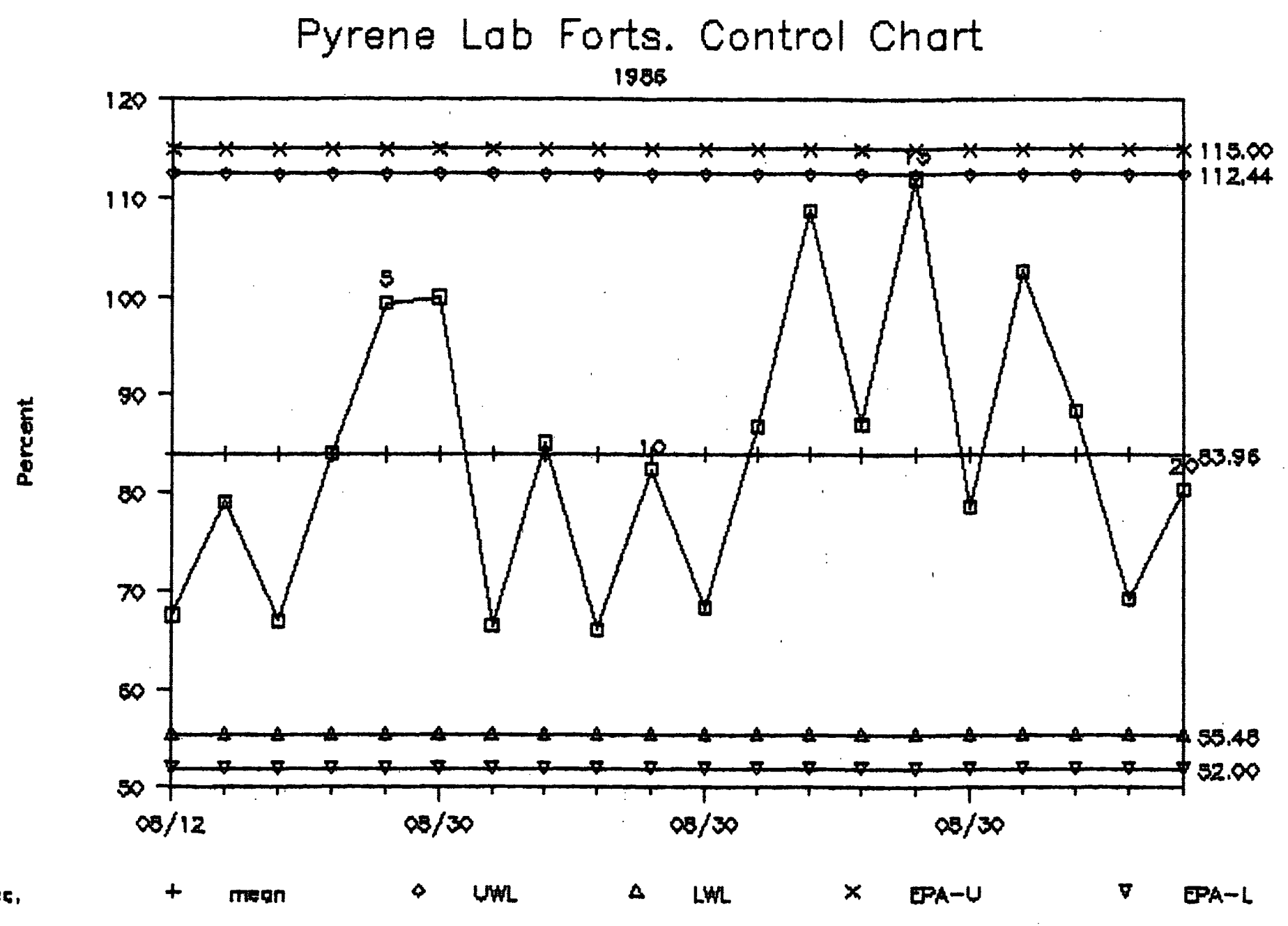
# Chrysene Surrogate Control Chart

1986



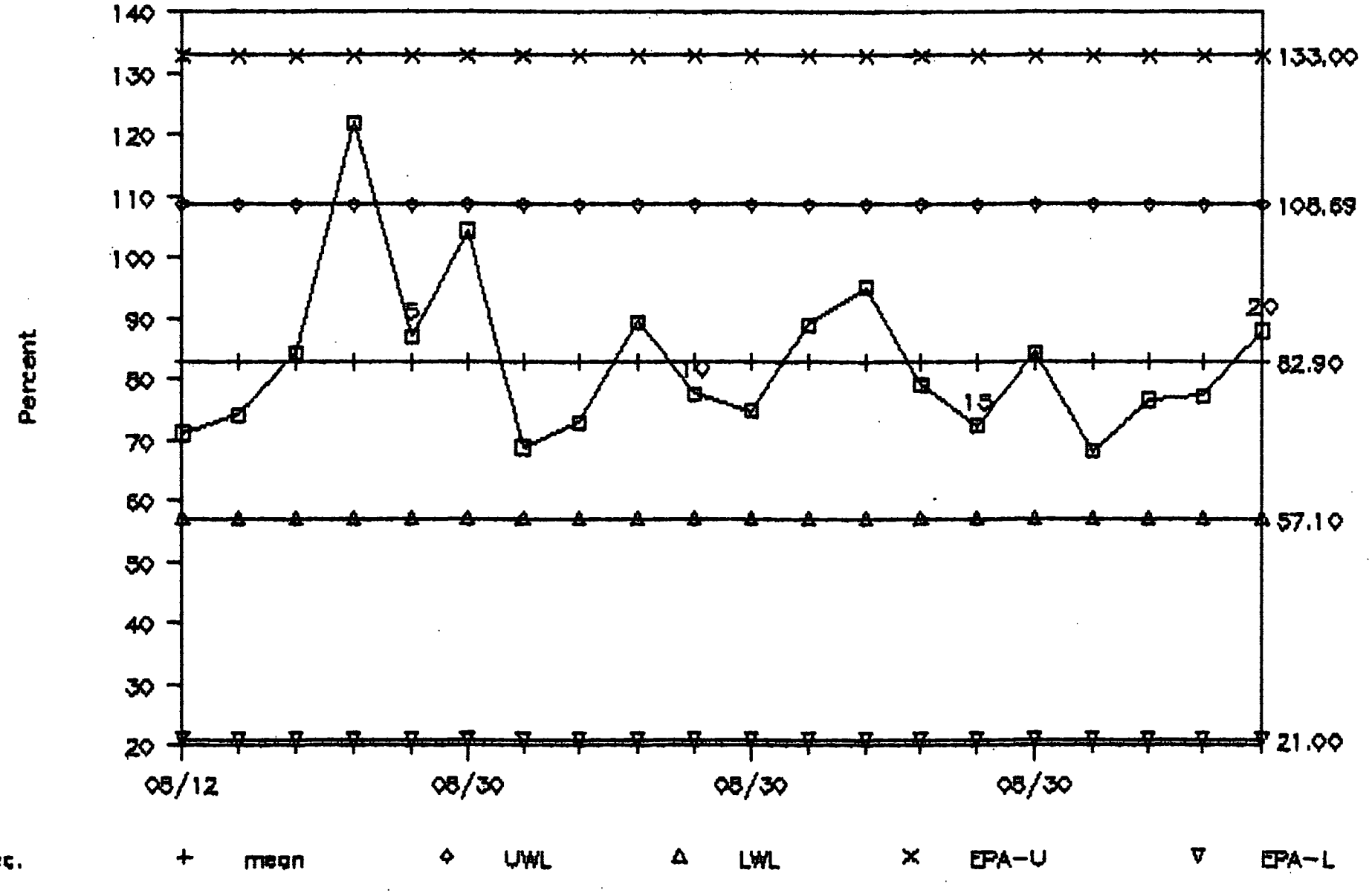
# Trichlrbenzene Lab Forts. Control Chart



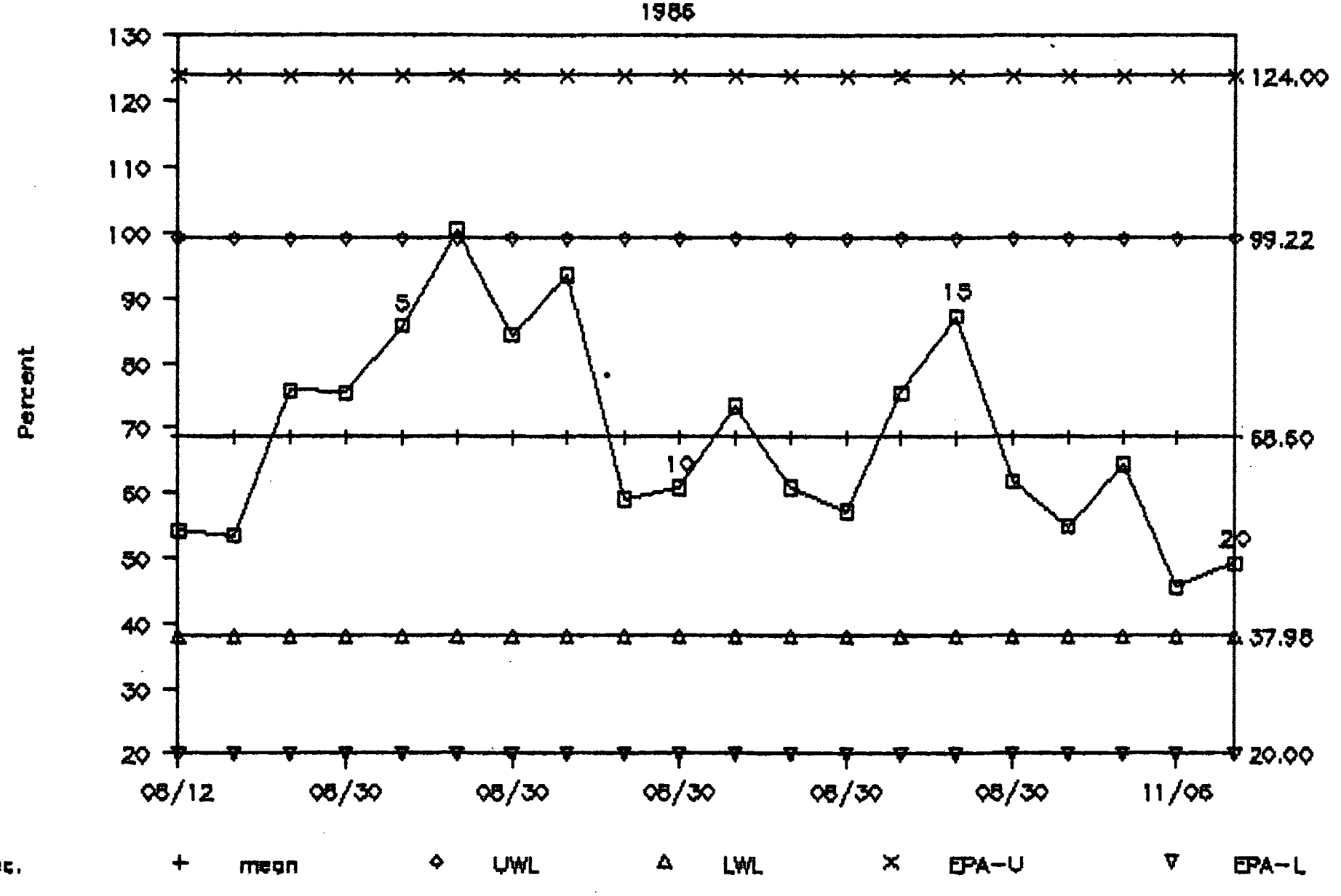

Pyrene Lab Forts. Control Chart
1986
Percent
120
110
100
90
80
70
60
50
115.00
112.44
83.96
55.48
52.00
05/12
05/30
05/30
05/30
5
10
15
20
+ mean
◇ UWL
△ LWL
× EPA-U
▽ EPA-L

# Naphthalene Lab Forts. Control Chart

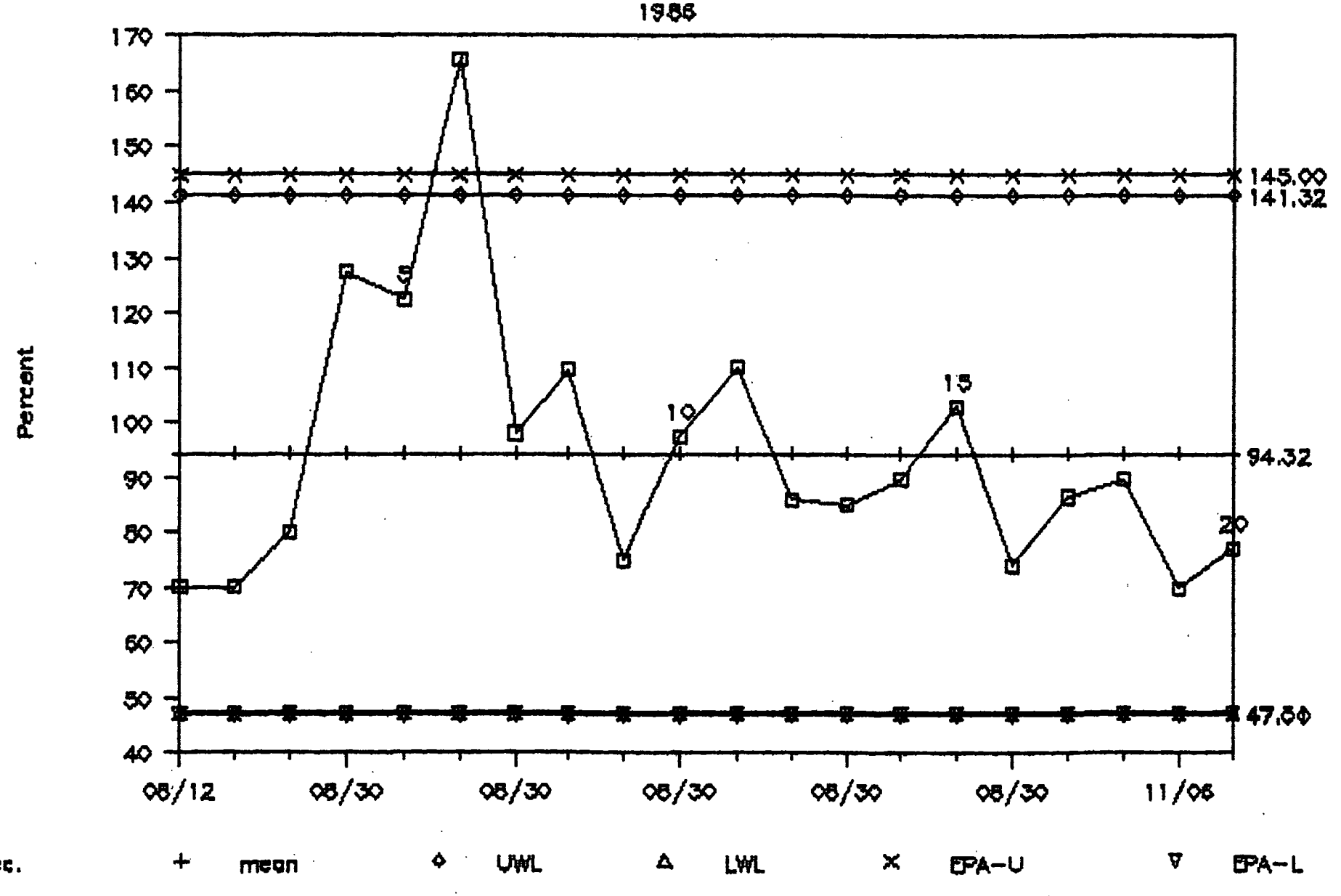
1986



# Dichlrobenzene Lab Forts. Control Chart

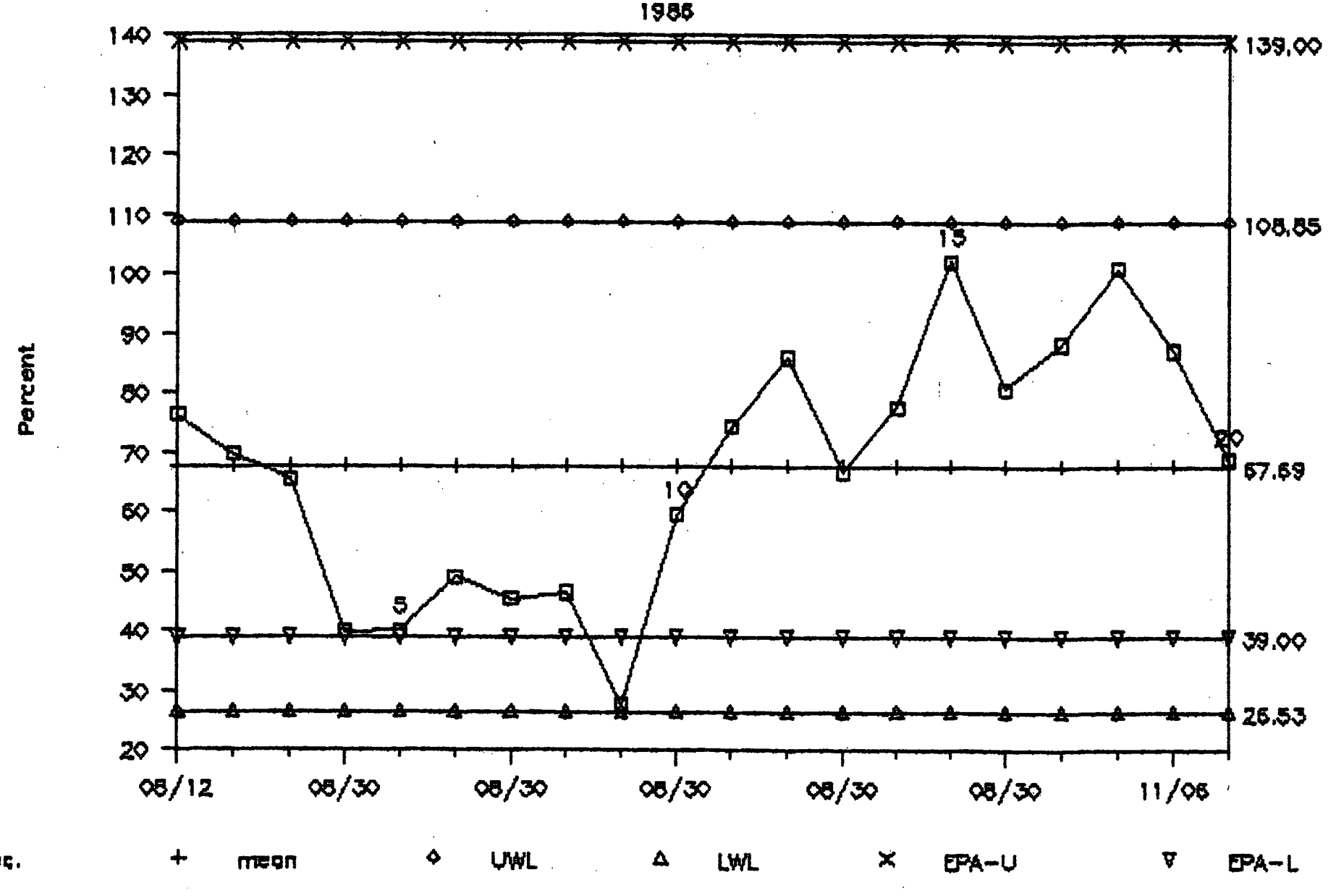


# Acenaphthene Lab Forts. Control Chart



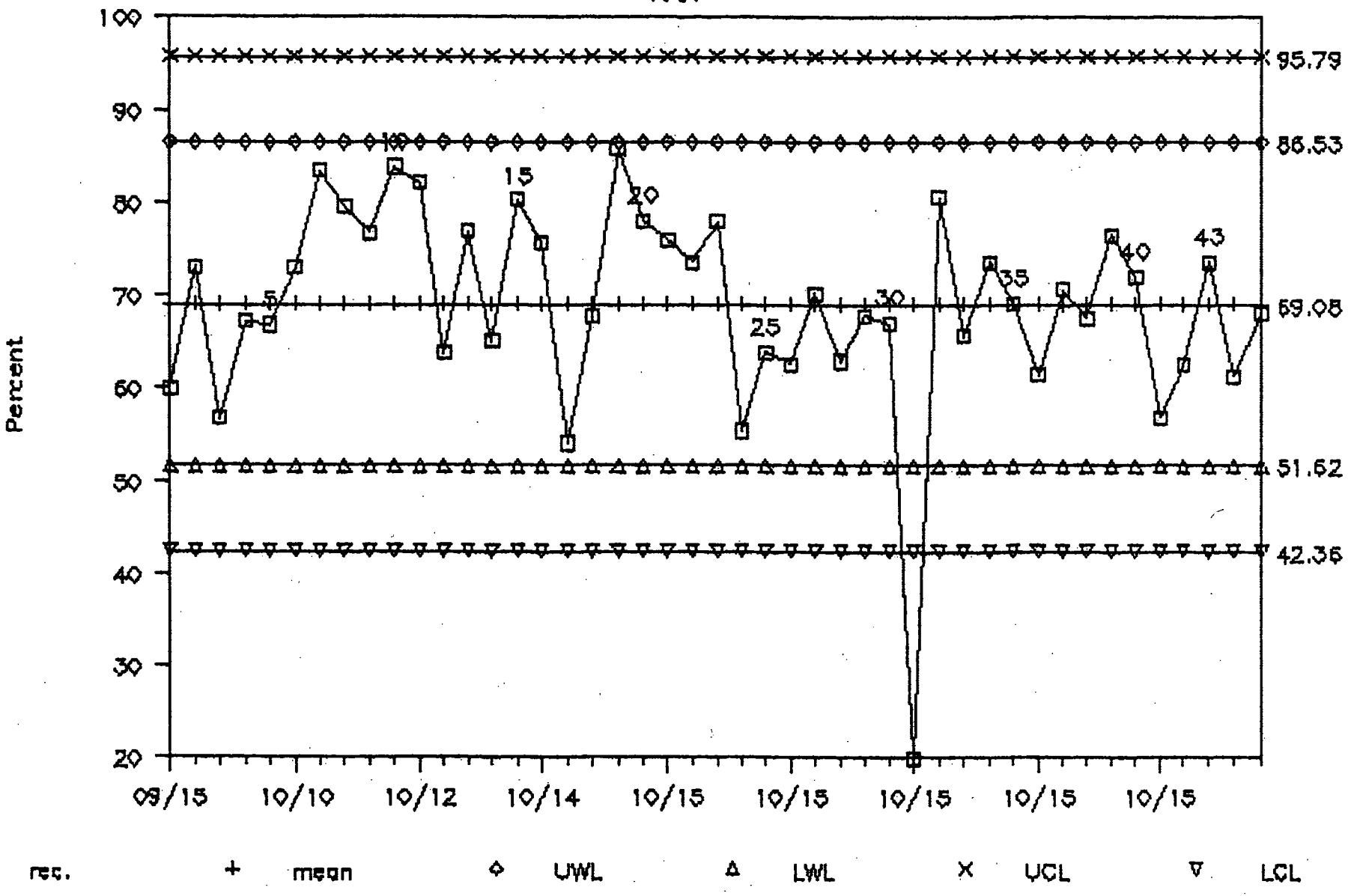

sc.

# Dinitrotoluene Lab Forts. Control Chart

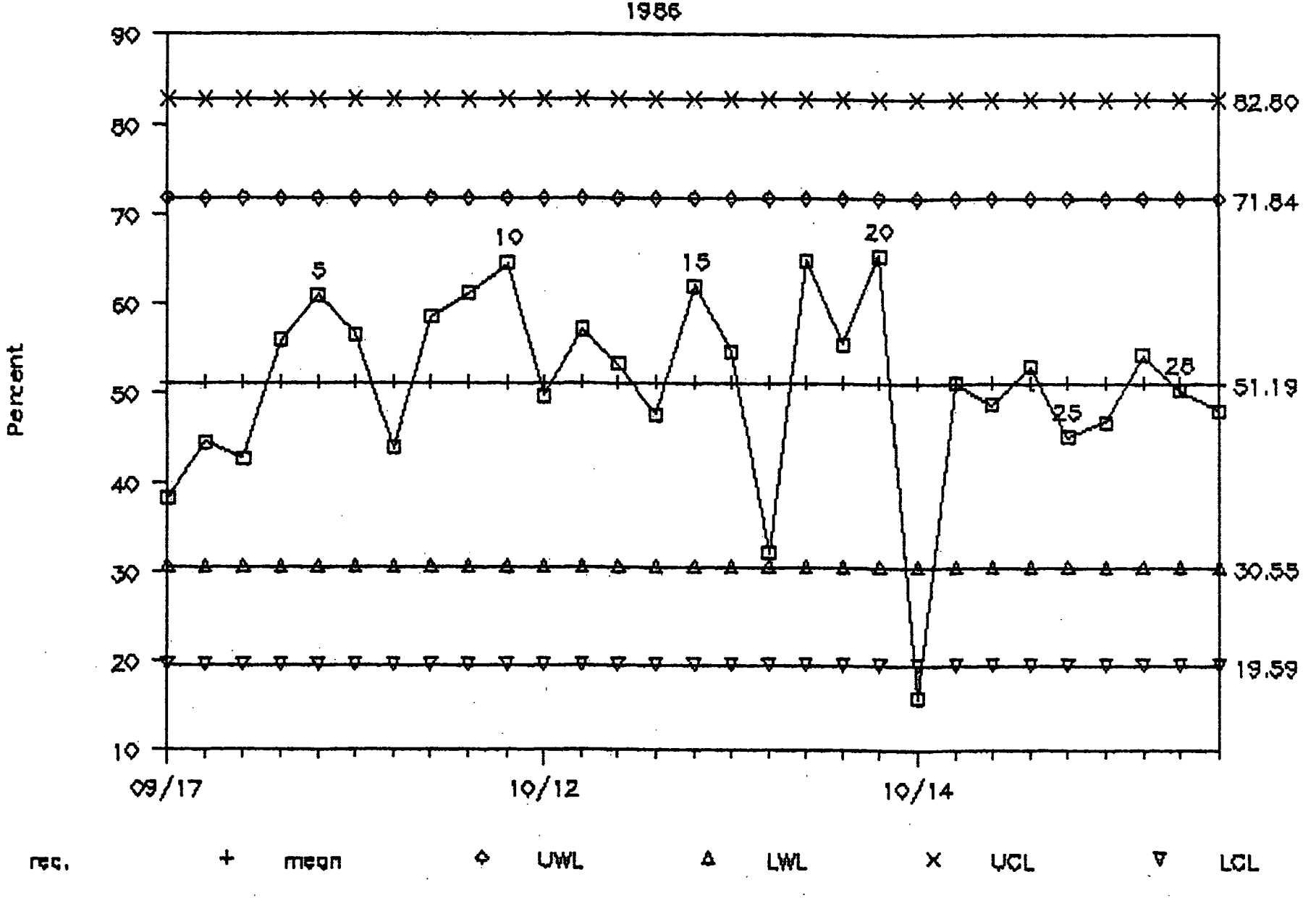


# Fluorobiphenyl Surrogate Control Chart

1986

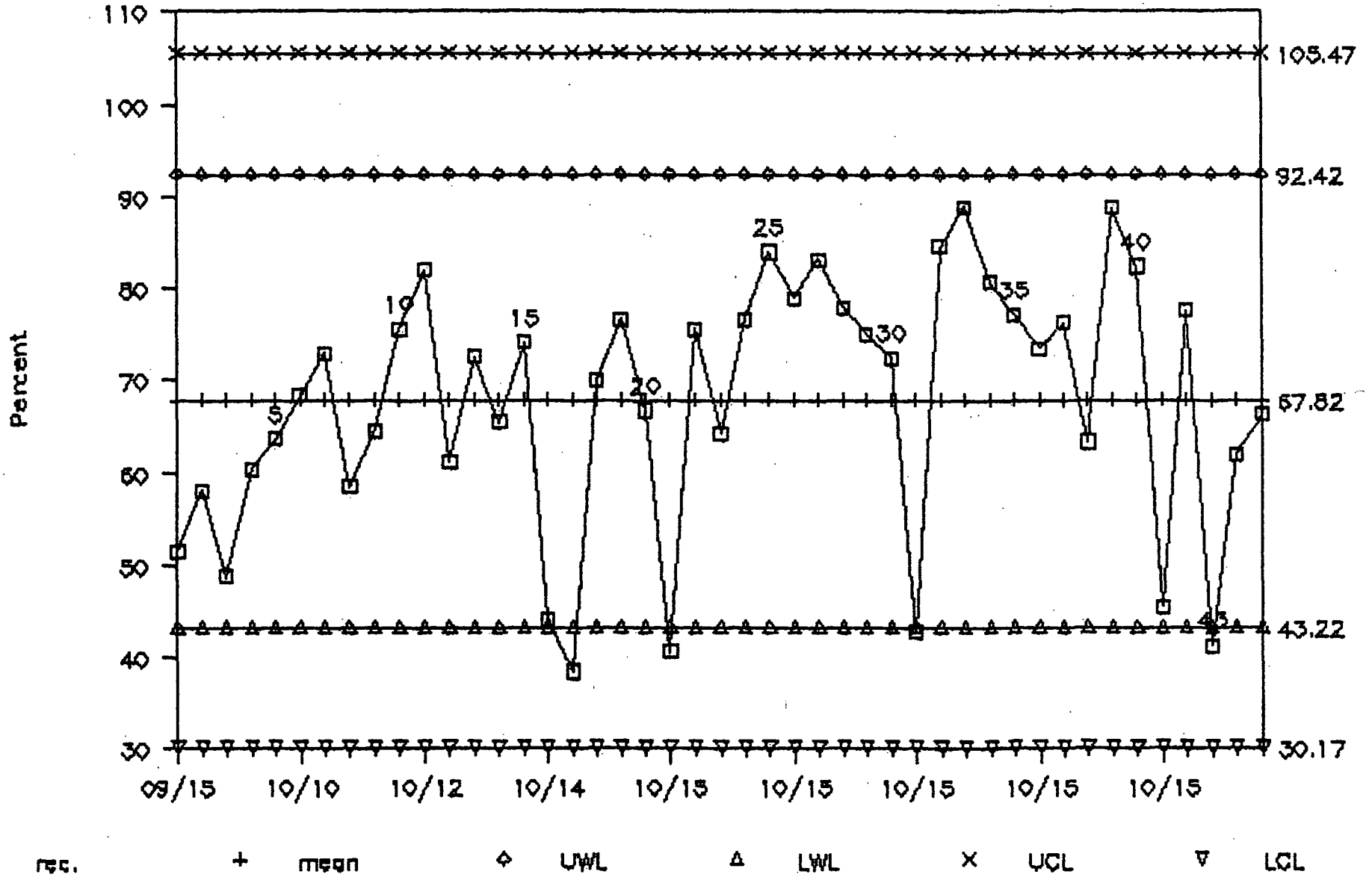


# 2—Fluorophenol Surrogate Control Chart



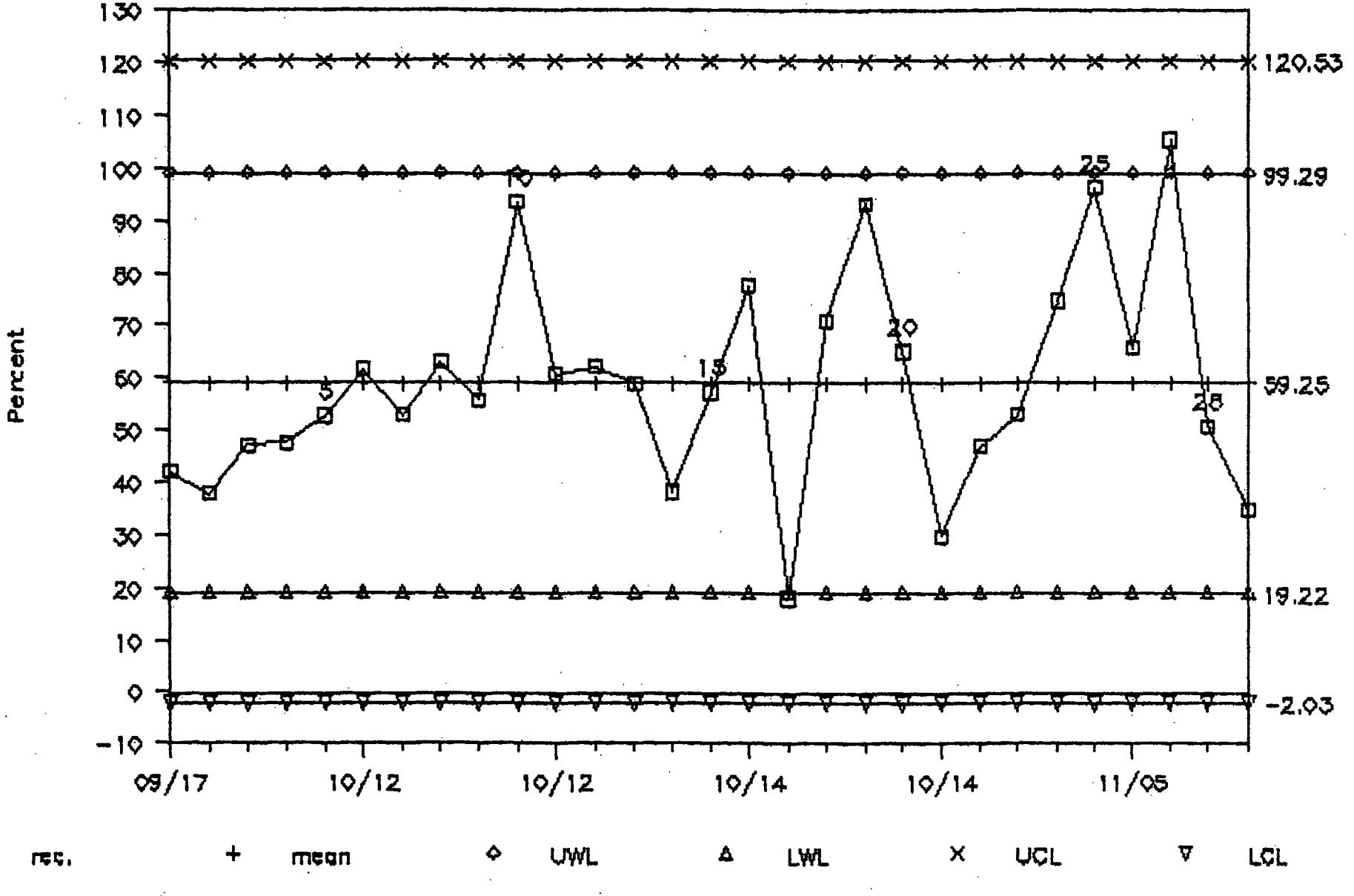
# Nitrobenzene Surrogate Control Chart

1986

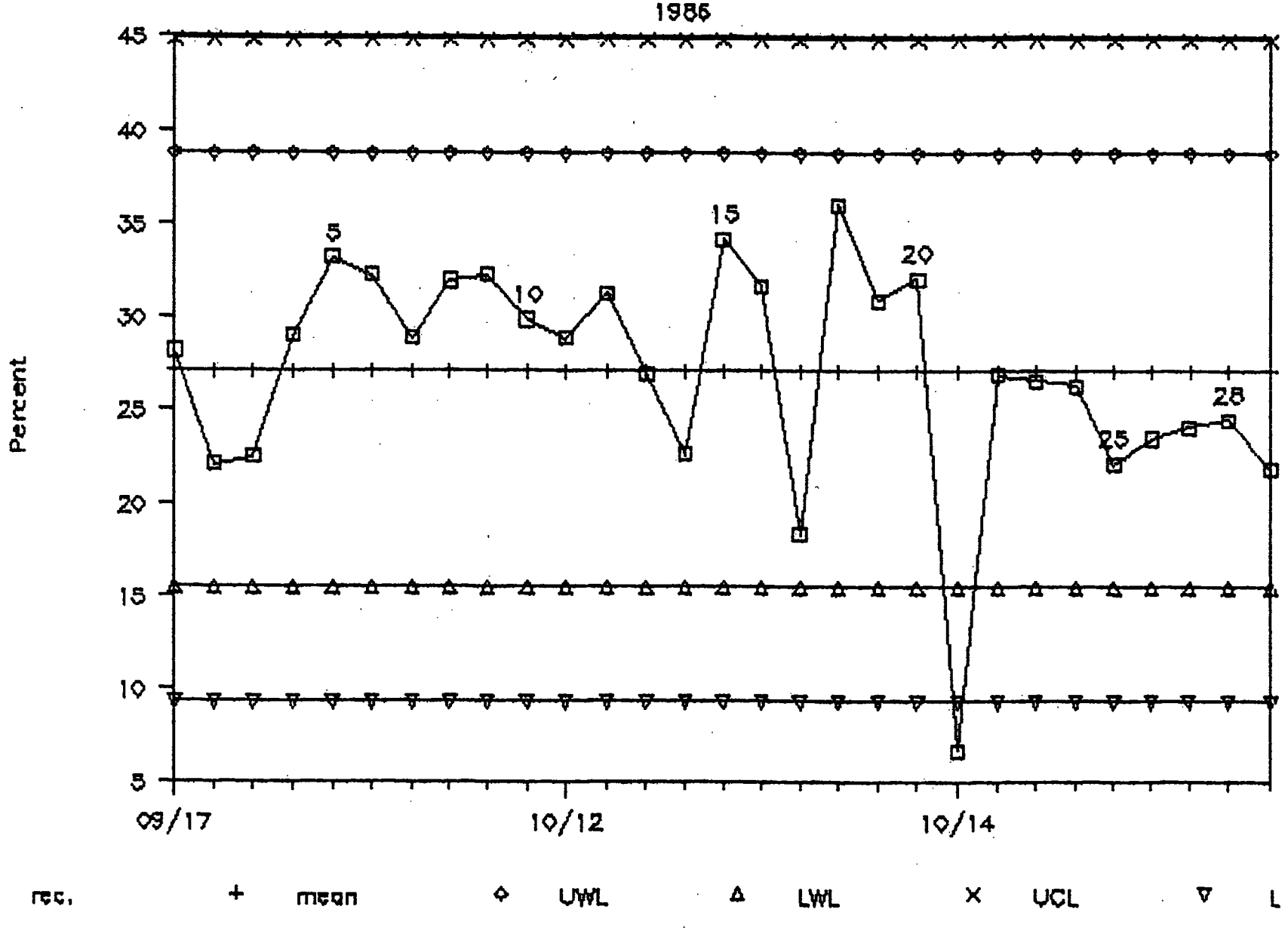


# Tribromophenol Surrogate Control Chart

1986



# Phenol-d5 Surrogate Control Chart



# Benzo(a)pyrene Surrogate Control Chart

1986

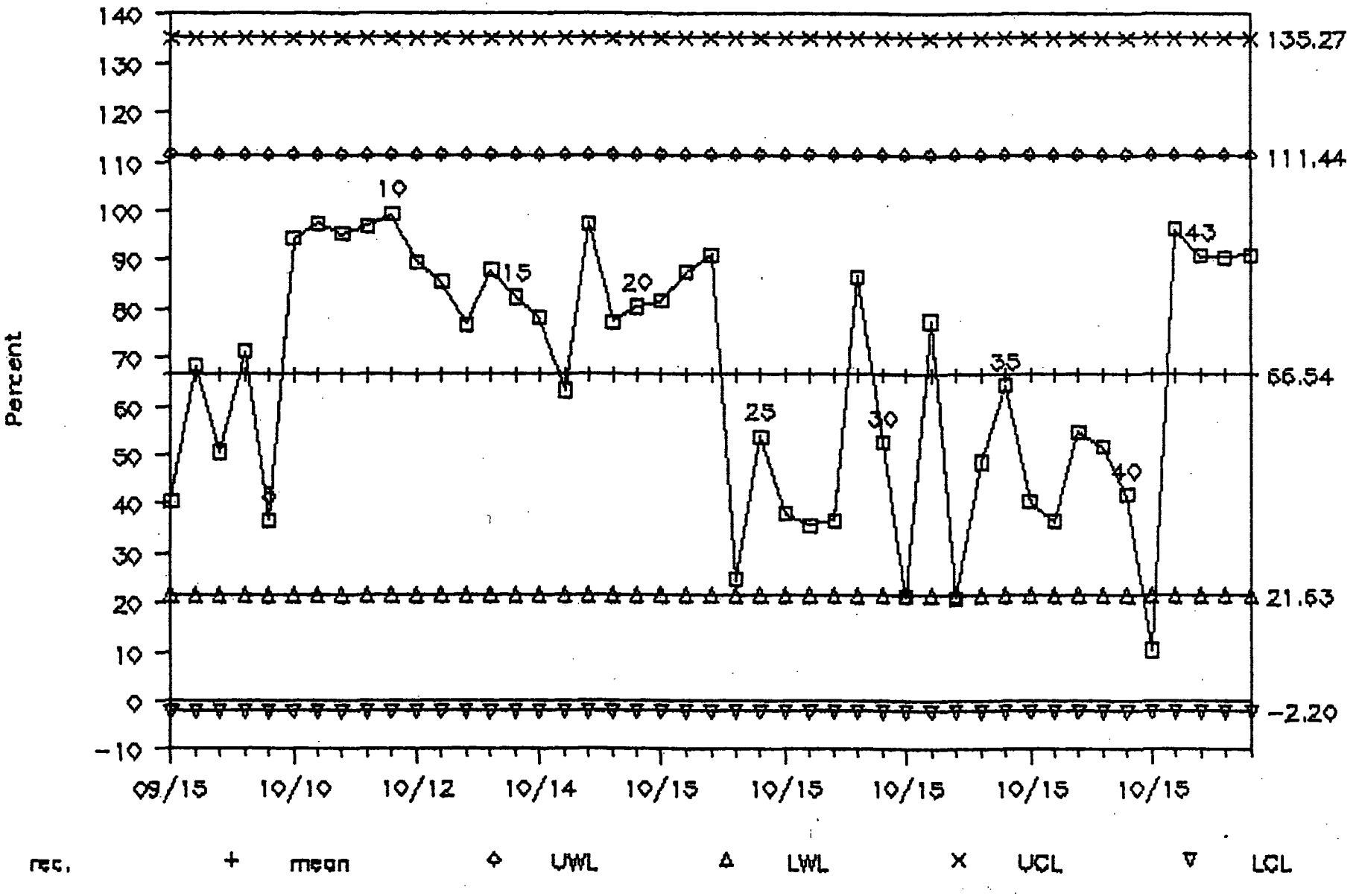


TABLE 5-1

STANDARD PAH AND OTHER PAH COMPOUNDS

FOR IDENTIFICATION AND QUANTITATION

a. Carcinogenic PAH

| Compound | Chemical Abstract Service Registry No. |
|---|---|
| benzo(a)anthracene | ( 56-55-3) |
| benzo(b)fluoranthene | (205-99-2) |
| benzo(j)fluoranthene | (205-82-3) |
| benzo(k)fluoranthene | (207-08-9) |
| benzo(ghi)perylene | (191-24-2) |
| benzo(a)pyrene | ( 50-32-8) |
| chrysene | (218-01-9) |
| dibenz(a,h)anthracene | ( 53-70-3) |
| indeno(1,2,3-cd)pyrene | (193-39-5) |
| quinoline | ( 91-22-5) |

b. Other PAH

| Compound | Chemical Abstract Service Registry No. |
|---|---|
| acenaphthene | ( 83-32-9) |
| acenaphthylene | (208-96-8) |
| acridine | (260-94-6) |
| anthracene | (120-12-7) |
| 2,3-benzofuran | (271-98-6) |
| benzo(e)pyrene | (192-97-2) |
| benzo(b)thiophene | ( 95-15-8) |
| biphenyl | ( 92-15-8) |
| carbazole | ( 86-74-8) |
| dibenzofuran | (132-64-9) |
| dibenzothiophene | (132-65-0) |
| 2,3-dihydroindene | (496-11-7) |
| fluoranthene | (206-44-0) |
| fluorene | ( 86-73-7) |
| indene | ( 95-13-6) |
| indole | (120-72-9) |
| 1-methylnaphthalene | ( 90-12-0) |
| 2-methylnaphthalene | ( 91-57-6) |
| naphthalene | ( 1-20-3) |
| perylene | (198-55-0) |
| phenanthrene | ( 85-01-08) |
| pyrene | (129-00-0) |

TABLE 4-4

GC RETENTION BEHAVIOR FOR PAH AND HETEROCYCLES

| Compound | M/Z | Retention Scan Number | SIM Sequence # |
|---|---|---|---|
| 2,3-benzofuran | 118 | 383 | 1 |
| 2,3-dihydroindene | 118 | 420 | 1 |
| Indene | 116 | 429 | 1 |
| Napthalene-d8 (Surr.) | 136 | 548 | 2 |
| Napthalene | 128 | 551 | 2 |
| Benzo(b)thiophene | 134 | 557 | 2 |
| Quinoline | 129 | 593 | 2 |
| Indole | 117 | 635 | 3 |
| 2-methylnapthalene | 141 | 640 | 3 |
| 1-methylnapthalene | 141 | 653 | 3 |
| Biphenyl | 154 | 703 | 3 |
| Acenaphthylene | 152 | 756 | 4 |
| Acenaphthene-d10 (IS-1) | 164 | 776 | 4 |
| Acenaphthene | 154 | 781 | 4 |
| Dibenzofuran | 168 | 802 | 4 |
| Fluorene-d10 (Surr.) | 176 | 843 | 4 |
| Fluorene | 166 | 848 | 4 |
| Dibenzothiophene | 184 | 956 | 5 |
| Phenanthrene-d10 (IS-2) | 188 | 970 | 5 |
| Phenanthrene | 178 | 974 | 5 |
| Anthracene | 178 | 980 | 5 |
| Acridine | 179 | 985 | 5 |
| Carbazole | 167 | 1004 | 5 |
| Fluoranthene | 202 | 1134 | 6 |
| Pyrene | 202 | 1162 | 6 |
| Benz(a)anthracene | 228 | 1333 | 7 |
| Chrysene-d12 (Surr.) | 240 | 1335 | 7 |
| Chrysene | 228 | 1339 | 7 |
| Benzofluoranthenes | 252 | 1496 | 8 |
| Benz(e)pyrene | 252 | 1536 | 8 |
| Benz(a)pyrene-d12 (IS-3) | 264 | 1539 | 8 |
| Benz(a)pyrene | 252 | 1543 | 8 |
| Perylene | 252 | 1546 | 8 |
| Indeno (1,2,3-cd)pyrene | 276 | 1713 | 9 |
| Dibenz(a,h)Anthracene | 278 | 1718 | 9 |
| Benzo(g,h,i)Perylene | 276 | 1750 | 9 |

TABLE 4-1

COMPOUNDS AND MS QUANTITATION MASS IONS

| Compound | Quantitation Mass Ion | Confirmation Ion (% Abundance) | Internal Standard Reference |
|---|---|---|---|
| **Polynuclear Aromatic Hydrocarbons (PAH)** | | | |
| Naphthalene | 128 | 102 (20) | 1 |
| Acenaphthylene | 152 | 151 (20) | 1 |
| Acenaphthene | 154 | 153 (90) | 1 |
| Fluorene | 166 | 165 (80) | 2 |
| Phenanthrene | 178 | 176 (20) | 2 |
| Anthracene | 178 | 176 (20) | 2 |
| Fluoranthene | 202 | 200 (20) | 2 |
| Pyrene | 202 | 200 (20) | 2 |
| Benzo(a)anthracene | 228 | 226 (20) | 3 |
| Chrysene | 228 | 226 (20) | 3 |
| Benzofluoranthenes | 252 | 250 (25) | 3 |
| Benzo(a)pyrene | 252 | 250 (25) | 3 |
| Indeno(1,2,3,cd)pyrene | 276 | 274 (20) | 3 |
| Dibenz(a,h)anthracene | 278 | 276 (20) | 3 |
| Benzo(g,h,i)perylene | 276 | 274 (20) | 3 |
| **Internal Standards** | | | |
| 1) Acenaphthene-d10 | 164 | | - |
| 2) Phenanthrene-d10 | 188 | | - |
| 3) Benz(a)pyrene-d12 | 264 | | - |
| **Surrogates** | | | |
| 1) Naphthalene-d8 | 136 | | 1 |
| 2) Flourene-d10 | 176 | | 2 |
| 3) Chrysene-d12 | 240 | | 3 |

TABLE 4-1 (Continued)

COMPOUNDS AND MS QUANTITATION MASS IONS

| Compound | Quantitation Mass Ion | Confirmation Ion (% Abundance) | Internal Standard Reference |
|---|---|---|---|
| Heterocycles and Other PAH | | | |
| Indene | 116 | 115 (90) | 1 |
| Indole | 117 | 90 (40) | 1 |
| 2,3-dihydroindene | 118 | 117 (50) | 1 |
| 2,3-benzofuran | 118 | 90 (40) | 1 |
| Quinoline | 129 | 102 (30) | 2 |
| Benzo(b)thiophene | 134 | -- | 2 |
| 2-methyl napthalene | 141 | 115 (40) | 2 |
| 1-methyl napthalene | 141 | 115 (40) | 2 |
| Biphenyl | 154 | 153 (30) | 3 |
| Carbazole | 167 | 166 (25) | 3 |
| Dibenzofuran | 168 | 139 (25) | 3 |
| Acridine | 179 | 178 (25) | 3 |
| Dibenzothiophene | 184 | 139 (20) | 3 |
| Perylene | 252 | 250 (30) | 3 |
| Benzo(e)pyrene | 252 | 250 (30) | 3 |
| Internal Standards | | | |
| 1) Acenaphthene-d10 | 164 | | - |
| 2) Phenanthrene-d10 | 188 | | - |
| 3) Benz(a)pyrene-d12 | 264 | | - |
| Surrogates | | | |
| 1) Naphthalene-d8 | 136 | | 1 |
| 2) Flourene-d10 | 176 | | 2 |
| 3) Chrysene-d12 | 240 | | 3 |

# INITIAL SAMPLING PLAN FOR THE REILLY TAR & CHEMICAL CORP. N.P.L. SITE ST. LOUIS PARK, MINNESOTA

October 4, 1986

ERT
A RESOURCE ENGINEERING COMPANY

# INITIAL SAMPLING PLAN FOR THE REILLY TAR & CHEMICAL CORP. N.P.L. SITE ST. LOUIS PARK, MINNESOTA

October 4, 1986

Prepared for

CITY OF ST. LOUIS PARK
St. Louis Park, MN

ERT - A RESOURCE ENGINEERING COMPANY
696 Virginia Road, Concord, Massachusetts 01742

8692P PE-415

CONTENTS

# SECTION A

# SITE MANAGEMENT PLAN

# INTRODUCTION

This Site Management Plan outlines the scope of work to be performed in order to monitor the ground water in the St. Louis Park, MN area in accordance with the Consent Decree - Remedial Action Plan (RAP) related to the Reilly Tar & Chemical Corp. N.P.L. site. Included in this plan are: 1) the identity of wells to be monitored, 2) the schedule for ground-water monitoring, and 3) a description of the procedures that will be used for sample collection, water level measurement, sample handling, sample analysis, and reporting.

The time period covered by the initial sampling plan is from the date of its acceptance and approval by the agencies, to December 31, 1987. This is one year longer than the initial plan is required to cover as stated in the RAP (section 3). The reason for this change is that, according to the schedule in the RAP, a sampling plan for 1987 would be due before comments were received on the initial sampling plan. Therefore, to avoid that situation, and to present a clear picture of ground-water monitoring activities through the first year of monitoring, this plan covers sampling through the 1987 calendar year. The first subsequent sampling plan (RAP section 3.3) will be submitted by October 31, 1987, covering the 1988 calendar year.

This plan incorporates the requirements of RAP Sections 3.2, 3.3, 4.3, 5.1, 6.1.4, 7.3, 8.1.3, 9.1.3, 9.2.3, 9.3.3, and 9.6. Some of the sampling required under RAP Section 4.3 (Monitoring the GAC System) has already taken place prior to the Effective Date. Therefore, only the monitoring that will take place from the approval date of this sampling plan through December 31, 1987 is included in this plan.

## SAMPLING SCHEDULE

The actual dates of ground-water monitoring are based on the timing of activities conducted under the RAP, and these dates cannot be predicted now with certainty. For example, except for the interim monitoring of the GAC plant, no monitoring will take place until this plan is approved. Therefore, the proposed sampling schedule outlined in this sampling plan indicates the starting criteria and the frequencies of sampling as outlined in the RAP to determine when the wells are sampled (Table 1). In general, the sampling schedule will allow economies of scale in the field and in the laboratory, by grouping the various monitoring events described by the RAP as much as possible. Samples will be collected within the time periods indicated on Table 1.

Table 1 summarizes the ground-water monitoring schedule for the period through December 1987. This table presents monitoring schedules for wells that have not been built yet (e.g. five new St. Peter Aquifer monitoring wells, RAP Section 8.1.3) and for wells that have not been retrofitted for long-term pumping (e.g., wells W23 and W105). The monitoring of these wells will begin during the sampling period covered in this plan, but the exact time is not certain. Subsequent progress reports, which are required under Part K of the Consent Decree, should be relied upon to provide better information on sampling dates for these wells. Also, all parties will be given two weeks notice in advance of routine sampling.

The duration of field sampling events will depend on the number and type of wells to be sampled. For estimating purposes, it is assumed that between 10 and 20 active pumping wells (e.g., municipal, industrial, or gradient/source control wells), and between 4 and 8 monitoring wells can be sampled in one day. It is a reasonable expectation that most sampling events will take place over the better part of a week, and some sampling may be done over a longer time frame.

### Identification of Wells to be Monitored

The RAP specifies the majority of wells to be monitored, but leaves the identification of 30 Drift-Platteville Aquifer wells to this plan. The 30 Drift-Platteville Aquifer wells identified in Table 1 (RAP Section 9.6) were selected to provide an adequate network for monitoring the distribution of PAH, and to evaluate the effectiveness of the source and gradient control well systems in the Drift-Platteville Aquifer. As such most of these wells are located down gradient, or on the periphery of the suspected area of contamination in the aquifer, and near the source and gradient control well systems. Also, the three new Drift Aquifer wells and three new Platteville Aquifer wells that will be

Which samples are to be analyzed for Cl, $SO_4$, $NH_3$, metals, etc?

TABLE 1. INITIAL SAMPLING PLAN GROUND-WATER MONITORING SCHEDULE [a]

| Source of Water | RAP Section | Sampling Points | Start of Monitoring | Sampling Frequency | Analyses |
|---|---|---|---|---|---|
| GAC Plant | 4.3.1 (C) | Treated water [b] | Date of plan approval | Monthly | PAH (ppt)[c] ✓ |
| | 4.3.3 (C) | Feed water | Date of plan approval | Quarterly | PAH (ppt) ✓ |
| Mt. Simon-Hinckley Aquifer | 5.1 | SLP11, SLP12, SLP13, SLP17, DW | Within six months of Effective date [g] | Annually | PAH (ppt) |
| Ironton-Galesville Aquifer | 6.1.4 | W105[e] | Start of pumping | Quarterly | PAH (ppb)[d] |
| Prairie du Chien-Jordan Aquifer | 7.3 (A) | SLP4 – DW | Start of pumping | Quarterly | PAH (ppt)[h] |
| | 7.3 (B) | W23 | Start of pumping | Quarterly | PAH (ppb) |
| | 7.3 (C) | SLP6, SLP7 or SLP9, W48 – DW | Date of plan approval | Quarterly | PAH (ppt) |
| | 7.3 (D) | AHM or MGC[i], E2, E13, H3, SLP10 or SLP15, SLP14, SLP16, W402[j] W403, W119 DW – | Date of plan approval | Semi-annually | PAH (ppt) |
| | 7.3 (E) | SLP5, H6, E3, E15, MTK6, W29, W40, W70, W401[j] | Date of plan approval | Annually | PAH (ppt) |
| | 7.3 (F) | W112, W32, SLP8, SLP10, E4, E7 | Date of plan approval | Quarterly | No chemical analyses [f] |
| St. Peter Aquifer | 8.1.3 | SLP3, W14, W24, W33, W122, W129 W133, P116, plus 5 new wells | Within 30 days of installing new wells | Once | PAH (ptt) |
| | | SLP3 plus six of the wells listed above | Within 6 months of above | Once | PAH (ppt) |

TABLE 1 (continued)

| Source of Water | RAP Section | Sampling Points | Start of Monitoring | Monitoring Frequency | Analyses |
|---|---|---|---|---|---|
| Drift-Platteville Aquifer | 9.1.3 and 9.2.3 | Source and gradient control wells (3 wells) | Start of pumping | Quarterly | PAH (ppb) and phenolics |
| | 9.3.3 | W131, W136, plus 6 new wells | Within 30 days of well installations | Semi-annually | PAH (ppb) and phenolics |
| | 9.6(k) | Drift: W2,W5 W6,W11,W12, W16, W116, W117, W128, W135, W136, PB140; Platteville: W1, W19, W20, W22, W115, W120, W121, W123, W130 W131, W132, W143, plus 6 new wells | Concurrent with 9.3.3 sampling | Concurrent with 9.3.3 sampling | PAH (ppb) and phenolics |

(a) This schedule does not include contingencies (eg. exceedance monitoring) and, therefore, represents the minimum program that is likely to occur between the date this plan is approved and December 31, 1987. The first samples will be collected during the period indicated by the monitoring frequency following the date of the start of monitoring.

(b) GAC plant treated water will be tested annually for extended PAH and acid fraction compounds as specified in RAP Section 4.3.4.

(c) ppt = parts per trillion. This signifies analysis using selected ion monitoring gas chromatography mass spectrometry.

(d) ppb = parts per billion. This signifies analysis by EPA Method 625. If analytical results for individual wells are below detection using this method, then the part per trillion method will be used on subsequent monitoring rounds.

(e) Water levels in W38 will be measured each time W105 is sampled.

(f) Water levels only (no monitoring) will be measured at these wells, except for those wells which prove to be inaccessible for such measurements.

(g) Or within 30 days of the approval date of this plan, whichever is later.

TABLE 1 (continued)

(h) SLP4 analytical program will be determined by the results of the feasibility study.

(i) AHM = American Hardware Mutual, MGC = Minikahda Golf Course.

(j) Wells W401, W402, and W403 may or may not be available for sampling at the same time as the other wells on these lists. They will be sampled in conjunction with the monitoring performed in accordance with the schedule shown, once they are available for sampling.

(k) If the six new Drift-Platteville Aquifer monitoring wells are not available for semi-annual sampling for the first year following the effective date, then monitoring of the wells listed here will be delayed in order to meet the RAP requirement of sampling these wells concurrently with the Northern Area Remedial Investigation (RAP Section 9.3.3).

If any of the wells listed here become damaged, destroyed, or otherwise unsuitable for sampling, alternate wells will be selected for monitoring.

installed for the Northern Area Remedial Investigation (RAP section 9.3.3) are included in the total of 30 wells.

The six St. Peter Aquifer wells that will be monitored under RAP section 8.1.3 will be selected based on the results of the first monitoring round.

## GROUND-WATER SAMPLING PROCEDURES

An important distinction is made between the sampling procedures for active pumping wells (eg. municipal wells) and for non-pumping monitoring wells. Active pumping wells are used on a regular basis, have dedicated pumps and associated plumbing, and have sample taps for collecting samples. Non-pumping monitoring wells may be new, or may have not been pumped for several years, and most require pumping and associated equipment for sampling. Another distinction is that the active pumping monitoring wells are typically located inside buildings whereas monitoring wells are not.

With these considerations in mind the sampling plan has been developed so that the ground-water monitoring program in each aquifer meets the requirements and intent of the RAP. Ground-water monitoring will be conducted in accordance with the procedures given in the Quality Assurance Project Plan (QAPP), and with "Procedures for Ground-Water Monitoring: Minnesota Pollution Control Agency Guidelines", April 1985.

### Sample Collection at Active Pumping Wells

At active pumping wells the sampling team will first determine that the wells have actually been pumping during the period preceding sampling. This information may be derived from inspecting flow recorders or from interviewing knowledgeable persons regarding the wells (water department employees, well owners, etc.). The information will be documented in the field notes of the sampling team.

Water level measurements will then be made, if practical. Sampling will proceed by filling the required containers with water from the sampling tap as near to the well head as possible, and before any holding tanks or treatment is encountered. The only exception to this is the GAC plant monitoring under RAP section 4.3 which includes treated-water monitoring.

If it can not be determined that a well has been pumping at some time during the 24 hour period preceding sampling, or if it is known the well was not pumping, then the well shall be pumped until field measurements of temperature, pH, and specific conductance have stabilized. These measurements, water levels, and the amount of water pumped will be recorded in the field notes.

Sample Collection at Non-Pumping Monitoring Wells

The vast majority of the non-pumping monitoring wells are constructed with a 4-inch diameter well casing. One of the proposed Drift Aquifer wells is a 1-1/4 inch piezometer and two of the proposed new St. Peter Aquifer monitoring wells may be 6-inch diameter wells. The 4-inch and 6-inch diameter wells will be purged with 3-3/4 inch diameter submersible pumps, while the piezometer will be purged with a peristaltic pump.

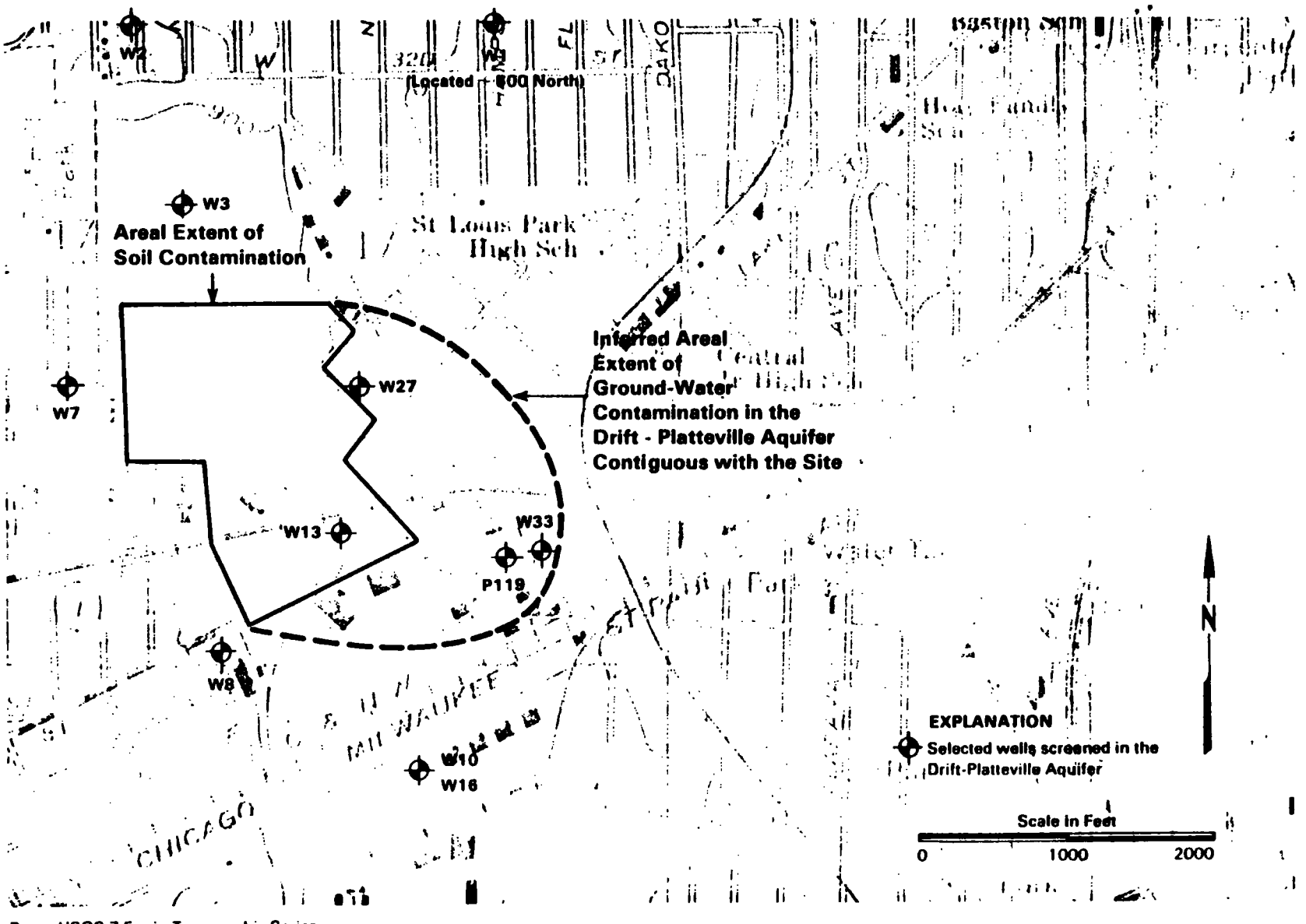
The general procedure at monitoring wells will be to first measure the water level and, for the initial sampling round, the depth of the well. The amount of water in one well volume will then be calculated.

The submersible pump will then be lowered into the well by hand using plastic-coated aircraft cable. Black plastic water pipe will be used for the discharge line. A plastic T-fitting will connect the black plastic line to a fire hose for routing the discharge to a sewer. Flexible silicon tubing with a clamp will be attached to the T-fitting and samples for field measurements will be collected through the tubing. A portable generator will be used to supply the power for the pump.

Each well will be purged prior to sample collection. The pump will be maintained in a position near the top of the water column to ensure proper purging of the well. Measurements of temperature, pH, and specific conductance will be made at intervals of one well volume until the values for these parameters, in three successive measurements, stabilize. Samples for PAH and/or phenolics will then be collected according to the procedures given in the QAPP. The QAPP also identifies the sample handling, quality control (field and trip blank schedule), and Chain of Custody procedures that will be followed during this program.

The discharge from purging monitoring wells will be routed to a sanitary sewer during the initial sampling event for each well. However, storm sewer or surface discharge will be used for monitoring wells located outside of the known area of contamination in the Drift-Platteville Aquifer, if the distance to the nearest sanitary sewer manhole is 200 feet or greater. There are probably less than five wells in this category. The area of Drift-Platteville contamination is depicted in Figure 1. Based on the analytical results of the first monitoring round, the discharge from subsequent well purgings will be routed to storm sewers using the criteria established in the RAP for PAH and phenolics. These criteria are:

| Parameter | Maximum Concentration Allowed in Surface Water Discharge |
|---|---|
| Other PAH | 34 parts per billion |
| Phenanthrene | 2 parts per billion |




Base: USGS 7.5 min Topographic Series
Hopkins and Minneapolis South Minnesota

Figure 1. Areal Limits of Soil and Surficial Ground-Water Contamination (ERT, 1983)

The black plastic water pipe and T-fitting with silicon tubing will remain with each well for use during subsequent sampling rounds. The submersible pump will be cleaned between wells for repeated use. The decontamination procedure will be to wash the pump throughly with soap and water. The pump will then be rinsed, and allowed to pump clean water for two to three minutes. St. Louis Park municipal water will be used for the decontamination procedure.

## ANALYTICAL PROGRAM

Table 1 shows the ground-water monitoring summary as prescribed in the RAP. Indicated on the table are the analyses required. Expanded analyses including some priority and conventional pollutants may also be required according to RAP Section 9.3.3. Details of all analytical methodology can be found in the QAPP. Organic analyses and metals analyses will be performed at ERT's Concord, Massachussetts laboratory facility. The Concord laboratory is the primary laboratory and all PAH and phenolics analyses will be performed at that location. The inorganic analyses will be performed at ERT's Houston, Texas laboratory facility. The laboratories have agreed to provide a turnaround time of 28 working days from the receipt of samples to the submittal of analytical reports. The laboratory will notify the City of St. Louis Park if it can not meet this turnaround time.

Ground-water monitoring will include two methods of PAH analyses depending upon the anticipated PAH concentration levels. Ultra-trace level (part per trillion) PAH analyses will be performed utilizing selected ion monitoring gas chromatography mass spectrometry. This method will be used to analyze samples from drinking water wells and from other wells for which the RAP requires drinking water criteria to be enforced (e.g., St. Peter Aquifer monitoring wells). Trace level (part per billion) PAH analyses, using the modified EPA Method 625, will be performed on samples from wells that have historically contained elevated PAH concentrations (e.g., part per million levels in wells W23 and W105), and on wells that are not subject to the RAP's requirements for meeting drinking water criteria (e.g., Drift-Platteville Aquifer monitoring wells).

Two methods are required for PAH analyses because the ultra-trace part per trillion method is not appropriate for samples containing in excess of approximately 1 part per billion PAH. Analysis of samples containing PAH concentrations over 1 part per billion, if performed with the ultra-trace method, requires mutiple dilutions and increases the risk of cross-contamination of the samples. This decreases the reliability of the data. Not only will multiple dilutions increase the variability of measurements, but critical quality control information (e.g., surrogate recoveries) is lost. Therefore, for samples containing greater than 1 part per billion PAH the analytical method, that is appropriate for the generation of reliable data is the modified EPA Method 625 as described in the Quality Assurance Project Plan (Section 4.6).

The modified EPA Method 625 analysis will be performed on two-liter samples, and will have detection limits of 1 part per billion. For wells that are tested with this method, if the analytical

which method will be used first? ultra trace or trace method.? ~~Basis~~ means of selection!

results of the first sampling indicates PAH concentrations less than 1 part per billion, the ultra-trace method will be used to analyze samples from subsequent sampling rounds. This procedure will allow an evaluation of long-term PAH concentrations around the fringe of PAH contamination in the Drift-Platteville Aquifer.

Depending on the circumstances and the actual PAH level, first-round analytical results using the ultra-trace method, that exceed 1 part per billion will indicate a switch to EPA Method 625 for subsequent sampling rounds.

## REPORTING

The analytical reporting requirements of the Consent Decree and RAP are identified in Part K of the Consent Decree, and Sections 3.4, 4.3.5, 12.1.1, and 12.1.2 of the RAP. Part K requires Reilly to submit quarterly progress reports on October 10, 1986, January 10, 1987, April 10, 1987, and July 10, 1987. These progress reports will contain analytical reports as specified in Section 5.0 of the QAPP for this initial sampling plan. The analytical results for samples collected in accordance with this initial sampling plan, but after the reporting period for the July 10, 1987 progress report, will be provided in next regularly scheduled progress report on March 15, 1988.

RAP Section 3.4 requires the City to submit an annual report that presents the results of all monitoring during the previous calendar year. The reports are due each March 15, 1987. The monitoring results that will be presented in the annual reports will include all water level measurements and chemical analyses. Interpretive maps and tables will be included in the annual reports, as specified in RAP Section 3.4(b) and (C). Also the effectiveness of the source and gradient control well systems inthe Drift-Platteville Aquifer will be discussed in the annual report.

RAP Section 4.3.5 requires the City to submit an annual report that presents the results of all monitoring of the GAC treatment system. Analytical results for wellhead water, feed water, and treated water will be included in this report. The report will also describe briefly the operating performance of the GAC plant during the previous calendar year. The GAC plant annual reports are due each March 15th, beginning in 1987.

RAP Sections 12.1.1 and 12.1.2 describe the re-sampling and analysis requirements in the event that samples from active municipal drinking water wells yield analytical results above advisory levels or drinking water criteria. Upon receipt of written analytical reports from the laboratory, the City will immediately notify the Regional Administrator, the Director, and the Commissioner of the exceedance. The notification will include the analytical data report as provided by the laboratory. The contents of these reports are discussed in detail in Section 5.0 of the QAPP for this initial sampling plan.

# SECTION B

# QUALITY ASSURANCE PROJECT PLAN

TABLE OF CONTENTS

# QUALITY ASSURANCE PROJECT PLAN
# FOR SAMPLING AND ANALYSIS - GROUNDWATER
# AND QAC PLANT MONITORING

Prepared for

The City of St. Louis Park
St. Louis Park, MN 55416

ERT, A Resource Engineering Company
696 Virginia Road, Concord, MA 01742

8692P PE-415

# 1. INTRODUCTION

## 1.1 Background

Groundwater in the City of St. Louis Park, Minnesota has been contaminated by activities at a coal-tar distillation and wood preserving plant operated from 1917 to 1972. A comprehensive report, prepared by ERT for Reilly Tar and Chemical Co., identified numerous polynuclear aromatic hydrocarbons (PAH) present in various aquifers beneath St. Louis Park and adjacent communities. Based upon extensive study of the migration, toxicity, and fate of PAH in the groundwater, ERT proposed a program of groundwater monitoring, treatment, and associated remedial measures, together with recommended water quality criteria for PAH, aimed at protecting the public health and reopening affected drinking water supply wells.

Through negotiations with the Environmental Protection Agency (EPA), the Minnesota Pollution Control Authority (MPCA), the Minnesota Department of Health (MDH), and St. Louis Park (SLP), acceptable water quality criteria were established. These criteria, incorporated into the final Federal Court Consent Decree, set the following concentration levels:

| | Advisory Level | Drinking Water Criteria |
|---|---|---|
| • Sum of benzo(a) pyrene and dibenz(a,h) anthracene | 3.0 ng/ℓ* | 5.6 ng/ℓ |
| • Carcinogenic PAH | 15 ng/ℓ | 28 ng/ℓ |
| • Other PAH | 175 ng/ℓ | 280 ng/ℓ |

*or the lowest concentration that can be quantified, whichever is greater.

In conjunction with the implementation of remedial measures to limit the spread of contaminants, a granular activated carbon (GAC) treatment system has been installed to treat water from St. Louis Park (SLP) wells 10 and 15. Further provisions of the Remedial Action Plan (RAP) call for long-term monitoring of the influent and effluent of the GAC treatment plant and the major aquifers underlying the region. This plan describes the Quality Assurance (QA) Quality Control (QC) criteria, audits and corrective action procedures pertaining to the sampling and analysis components of the monitoring program. Details of the sampling locations and schedules are provided in the Site Management Plan.

## 1.2 Quality Objectives

The principal objectives of this plan pertain to the collection of data that are sufficient to monitor the effectiveness of the GAC treatment system and to detect changes in groundwater quality. Therefore, the quality of the data gathered in this project can be defined in terms of the following elements:

- Completeness - a sufficient number of successful (valid) measurements to characterize the concentrations of PAH in the influent and effluent of the treatment system and in the aquifers of interest over a period of time.
- Representativeness - the extent to which reported analytical results truely depict the PAH concentrations in the sampled environment. Representativeness is optimized through proper selection of sampling sites, times and procedures, through proper sample preservation, and through prompt extraction and analysis.
- Accuracy and Precision - Accurate and precise data will be achieved through the use of sampling and analytical procedures that minimize biases, through the use of standard procedures, through the meticulous calibration of analytical equipment and by implementing corrective action whenever measured accuracy and precision exceed pre-established limits. Accuracy and precision will be measured by the analysis of method spikes and duplicate samples.
- Comparability - the extent to which comparisons among separate measurements will yield valid conclusions. Comparability among measurements in the SLP monitoring program will be achieved through the use of rigorous standard sampling and analytical procedures.
- Traceability - the extent to which results can be substantiated by hard-copy documentation. Traceability documentation exists in two forms: that which links final numerical results to authoritative measurement standards, and that which explicitly describes the history of each sample from collection to analysis.

This plan describes the procedures that will be implemented to ensure quality as defined above.

## 1.3 Project Organization and Responsibilities

The project organization is illustrated in Figure 1-1. The ERT Quality Assurance Department is completely independent of line function. Its manager reports directly and exclusively to the ERT Executive Vice President. Members of the QA Department are called Quality Assurance Officers. The Laboratory Quality Control Coordinator is appointed by the

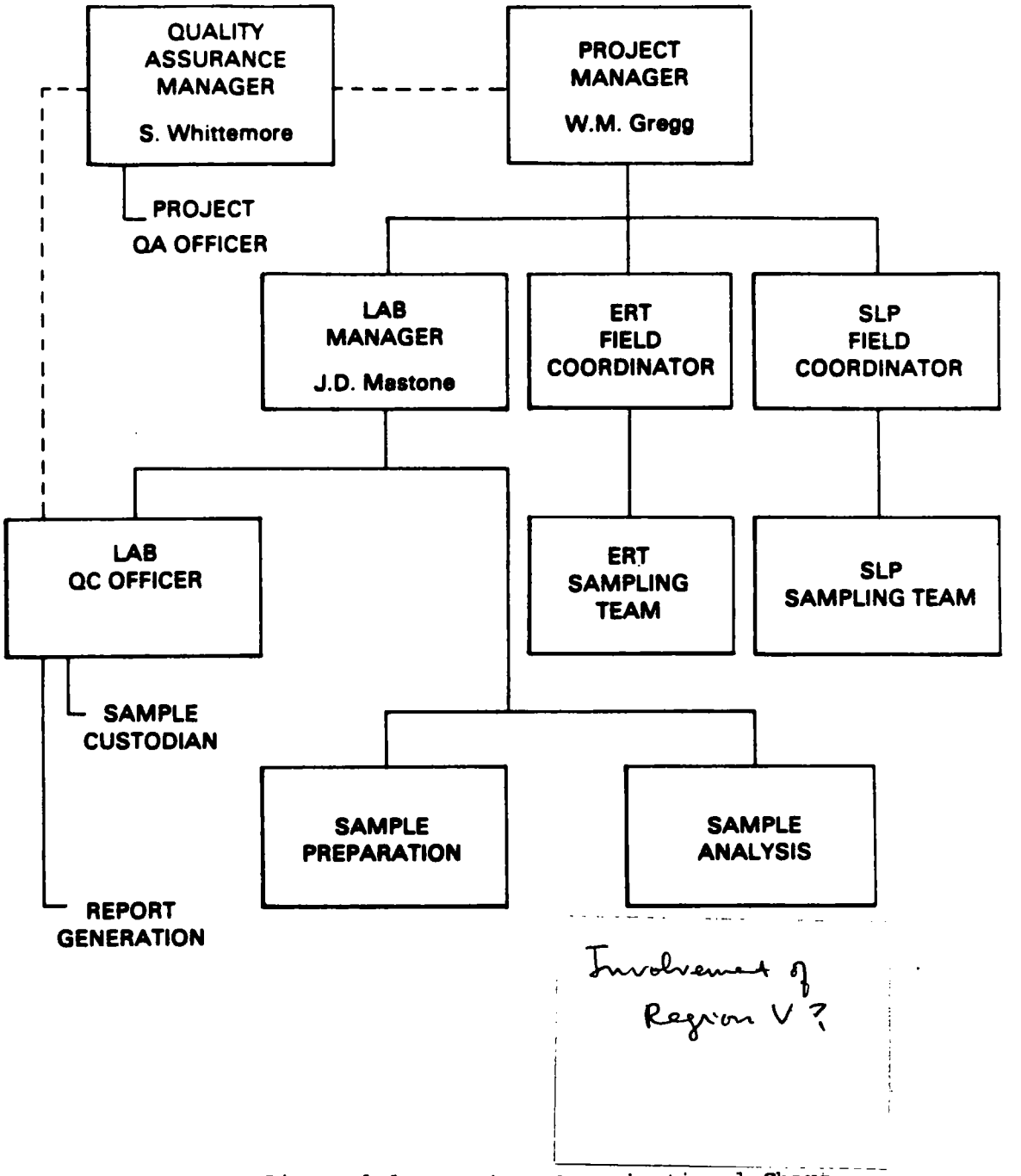



Figure 1-1 Project Organizational Chart

Chemistry Division Manager and reports directly to the Division Manager, with ancillary responsibilities to the Laboratory Manager and the Corporate Quality Assurance Manager.

All other functions in the organizational structure report directly through line management. Responsibilities of the key positions in the organization are described below:

- Project Manager: The Project Manager's responsibilities include correspondence, review of all project data, scheduling of activities, and authorization of revisions to the Sampling Plan and the QA Plan.
- Laboratory Manager: The Laboratory Manager is responsible for overall management of laboratory operations to meet project commitments, including scheduling of personnel and physical resources.
- Laboratory QC Coordinator: The Laboratory QC Coordinator is responsible for maintaining the laboratory Quality Control program. The Laboratory QC Coordinator maintains laboratory standards and traceability documentation and performs analytical data package validation. The Laboratory QC Coordinator reports directly to the Laboratory Manager, but also has indirect reporting responsibility to the Quality Assurance Manager.
- Laboratory Section Supervisor: The Laboratory Section Supervisor is responsible for supervising all aspects of sample preparation and analysis performed by analysts and technicians.
- Sample Custodian: The Sample Custodian is responsible for issuance of sampling kits to the Field Coordinator and for inspection and log-in of incoming samples and control of sample storage.
- Report Coordinator: The Report Coordinator is responsible for the review of raw data and corresponding data tables, compiling summary tables of sample results and corresponding QA/QC, and preparation of rough draft reports for review and comments by supervisors and managers.
- Field Coordinator: The Field Coordinator is responsible for the coordination and effective use of all personnel on site and will maintain a general log of activities. The Field Coordinator will also be responsible for issuance and tracking of measurement and test equipment, the proper labeling, handling, storage, shipping, and chain of custody procedures used at the time of sampling, and control and archiving of all field documentation, (log books, notebooks, data sheets, etc.) generated during the field investigation.
- Sampling Geologists/Engineers: The Sampling Geologists/Engineers responsibilities include collecting samples; conducting field

Responsibility for
1) Sample Collection
2) field QC ?

measurements; (e.g. water level) and maintaining proper decontamination procedures; all according to documented procedures stated in the Quality Assurance Plan and the corresponding SOPs.

- Analyst: The Analyst is responsible for the analysis of water samples for the requested parameters utilizing the methods prescribed by this plan.
- Technician: The Technician is responsible for sample extraction (according to documented procedures stated in Section 4.4). This requires practical experience and knowledge in the techniques of liquid--liquid solvent extraction, Kuderna Danish evaporation, and the quantitative preparation of sample extracts for analysis.

## 2. SAMPLING

Samples will be collected by ERT and SLP personnel. The overall sampling program is summarized in Table 2-1. Further details of sampling locations and frequency are provided in the Site Management Plan. This section discusses general QA provisions relevant to sample collection, containerization, packaging and shipping activities.

### 2.1 Training

All ERT and SLP personnel working on the project will be properly trained, qualified individuals. Prior to commencement of work, personnel will be given instruction specific to this project, covering the following areas:

- Organization and lines of communication and authority
- Overview of the Work Plan and QA plan,
- Documentation requirements,
- Decontamination requirements,
- Health and Safety considerations.

Training of field personnel will be provided by the Field Coordinator or his/her qualified designee.

The analysts performing chemical analyses of samples will be trained in and will have exhibited proficiency at the analytical methods to be employed.

### 2.2 Document Control

Document Control for the SLP Monitoring Program serves a two-fold purpose. It is a formal system of activities that ensures that:

1) All participants in the project are promptly informed of revisions of the Quality Assurance Plan; and

2) All critical documents generated during the course of the response actions are accounted for during, and at the end of the project.

This QA Plan and all Standard Operating Procedure documents have the following information on each page:

Sampling

TABLE 2-1

SUMMARY OF SAMPLING PROGRAM

| Source | Sampling Point | Sampling Frequency | Data Required (a) |
|---|---|---|---|
| GAC Treatment Plant | Effluent | Monthly | PAH (PPT)(b) |
| GAC Treatment Plant | Influent | Quarterly | PAH (PPT) |
| Mt. Simon-Hinkley Aquifer | SI.P11, SLP12, SLP13, SLP17 | Monthly (first 6 mo)<br>Annually (after 6 mo) | PAH (PPT)<br>PAH (PPT) |
| Ironton-Galesville Aquifer | W105 | Quarterly (for 1 year)<br>Annually (after 1 year) | PAH (PPB)(e)<br>PAH (PPB) |
| | W38 | Same as W105 | Water Level |
| Prairie du Chien-Jordan Aquifer | W23 | Quarterly (for 1 year)<br>Semiannually (after 1 year) | PAH (PPB)<br>PAH (PPB) |
| | SLP4 | Quarterly (for 1 year)<br>Semiannually (after 1 year) | PAH (PPT)(f)<br>PAH (PPT) |
| | W48, SLP6, SLP7 or SLP9, | Quarterly | PAH (PPT) |
| | AHM or MGC(c), E2, E13 H3, SLP10 or SLP15, SI.P14, SLP16, W402, W403, W119 | Semi-annually | PAH (PPT)(d) |
| | SI.P5, H6, R3, R15, MTK6, W29, W40, W70, W401 | Annually | PAH (PPT) |
| St. Peter Aquifer | SI.P3, W14, W24, W33, W122, W129, W133, P116, plus 5 new wells | Once, within 30 days of new well installation | PAH (PPT) |
| | SI.P3, plus six of the wells listed above | once six months after above, once twelve months after above | PAH(PPT) |
| Drift-Platteville Aquifer | Source and gradient Control wells | Quarterly | PAH (PPB), Phenolics |
| | W131, W136, plus six new wells | Once, within 30 days of new well installation and once within six additional months | PAH (PPB), Phenolics |
| | 30 wells | same as above | PAH (PPB), Phenolics |

(a)Water levels will be measured each time samples are collected.
(b)GAC plant treated water will be tested annually for the extended list of PAH and acid fraction compounds, as specified in RAP Section 4.3.4.
(c)AHM = American Hardware Mutual; MGC = Minikahda Golf Course.
(d)Water levels will be measured quarterly in wells W112, W32, SI.P8, SI.P10, E4, and E7 during this time period.
(e)If PPB analysis indicates no detectable PAH, PPT analysis will be performed thereafter.
(f)SI.P4 analytical program will be determined by the results of the feasibility study.

acid fraction at GAC plant ?

- Document Number
- Page Number
- Total number of pages in document
- Revision number
- Revision date

When any of these documents are revised, the affected pages are reissued to all personnel listed as document holders with updated revision numbers and dates. Issuance of revisions is accompanied by explicit instructions as to which documents or portions of documents have become obsolete.

Control of, and accounting for documents generated during the course of the project is achieved by assigning the responsibility for document issuance and archiving. Table 2-2 lists the key documentation media for the project and corresponding responsible parties for issuance, execution and archiving.

Table 2-3 is a list of ERT Standard Operating Procedures applicable to the project. The Standard Operating Procedures themselves are contained in Appendix A.

## 2.3 Sample Control Procedures and Chain of Custody

In addition to proper sample collection, preservation, storage and handling, appropriate sample identification procedures and chain of custody are necessary to help insure the validity of the data.

### 2.3.1 Sample Identification

Sample labels shall be completed for each sample, using waterproof ink, unless prohibited by weather conditions. For example, a logbook notation would explain that a pencil was used to fill out the sample tag because a ballpoint pen would not function in freezing weather. The information recorded on the sample label includes:

Station Location - Description of place where sample was taken (e.g. the well or sampling point indentification).

Date - A six-digit number indicating the year, month and day of collection.

Time - A four-digit number indicating the military time of collection.

Sampler - Signature of person collecting the sample.

Remarks - Any pertinent observations or further sample description.

After collection, identification, and preservation, the sample is maintained under chain-of-custody procedures discussed below.

TABLE 2-2
DOCUMENT CONTROL

| Item | Issued By | Issued To | Archived By |
|---|---|---|---|
| Field Notebooks | Field Coordinator | Sampling Team | Field Coordinator |
| Equipment Calibration Forms | Field Coordinator | Equipment Operators | Field Coordinator |
| Sample Logs | Field Coordinator | Sampling Team | Field Coordinator |
| Chain-of-Custody Forms | Lab Sample Custodian | Field Coordinator | Lab Sample Custodian |
| Sample Labels | Field Coordinator | Sampling Team | Lab Sample Custodian |

TABLE 2-3

ERT STANDARD OPERATING PROCEDURE LIST

| Name | Title |
|---|---|
| 7130 | Groundwater Sampling |
| 7230 | Operation of Hydrolab |
| 7510 | Packaging and Shipment of Samples |
| 7600 | Decontamination of Equipment |

2.3.2 Chain-of-Custody Procedures

To maintain and document sample possession, chain-of-custody procedures are followed. A sample is under custody if:

- It is in your possession, or
- It is in your view, after being in your possession, or
- It was in your possession and then you locked it up to prevent tampering, or
- It is in a designated secure area.

TRANSFER OF CUSTODY AND SHIPMENT

1. Samples are accompanied by a Chain-of-Custody Record (Figure 2-1). When transferring the possession of samples, the individuals relinquishing and receiving will sign, date, and note the time on the record. This record documents sample custody transfer from the sampler, often through another person, to the analyst at the laboratory.

2. Minimum information recorded on the chain-of-custody record in addition to the signatures and dates of all custodians will include:

   - Sampling site indentification
   - Sampling date and time
   - Chain-of-custody tape number
   - Identification of sample collector
   - Sample identification
   - Sample description (type and quantity)
   - Analyses to be performed.

3. Samples will be packaged properly for shipment and dispatched to the appropriate laboratory for analysis, with a separate custody record accompanying each shipment. Shipping containers will be sealed for shipment to the laboratory. Before sealing each container, select two pieces of chain-of-custody tape and enter their numbers on the chain-of-custody form (in your "relinquished

Figure 2-1

CHAIN OF CUSTODY RECORD

| Client/Project Name | Project Location | ANALYSES |
|---|---|---|
| Project No | Field Logbook No | |
| Sampler: (Signature) | Chain of Custody Tape No | |

| Sample No / Identification | Date | Time | Lab Sample Number | Type of Sample | | | | | | | | REMARKS |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | | | | | | | | |
| | | | | | | | | | | | | |
| | | | | | | | | | | | | |
| | | | | | | | | | | | | |
| | | | | | | | | | | | | |
| | | | | | | | | | | | | |
| | | | | | | | | | | | | |

| Relinquished by (Signature) | Date | Time | Received by (Signature) | Date | Time |
|---|---|---|---|---|---|
| Relinquished by (Signature) | Date | Time | Received by (Signature) | Date | Time |
| Relinquished by (Signature) | Date | Time | Received for Laboratory (Signature) | Date | Time |
| Sample Disposal Method | Disposed of by (Signature) | | | Date | Time |

SAMPLE COLLECTOR

Environmental Research and Technology, Inc.
696 Virginia Road
Concord, MA 01742
617-369-8810

ANALYTICAL LABORATORY

ERT

Nº 10501

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by" signature box). The method of shipment, courier name(s) and other pertinent information are entered in the "Remarks" box. Then tear off the last copy of the form and place the original and remaining copies in the container. After the container is sealed, place the chain-of-custody tape over the seal on opposite corners of the container.

4. Whenever samples are split with another laboratory, it is noted in the "Remarks" section. The note indicates with whom the samples are being split and is signed by both the sampler and recipient. If either party refuses a split sample, this will be noted and signed by both parties. The person relinquishing the samples to the facility or agency should request the signature of a representative of the appropriate party, acknowledging receipt of the samples. If a representative is unavailable or refuses to sign, this is noted in the "Remarks" space. When appropriate, as in the case where the representative is unavailable, the custody record should contain a statement that the samples were delivered to the designated location at the designated time.

### 2.3.3 Field Forms

In addition to sample labels and chain-of-custody forms, a bound field notebook must be maintained by the sample team leader to provide a daily record of significant events. All entries must be signed and dated. All members of the of the sampling team must use this notebook. Keep the notebook as a permanent record.

## 2.4 Sampling Procedures - GAC Plant

All sampling will be performed under the direction of the St. Louis Park Water Department personnel. All sampling materials will be provided by ERT, including bottles, labels, tapes, shipping containers and chain-of-custody forms.

Samples should be collected at a convenient time during the normal working day. Samples scheduled for the same day will be collected in close succession, moving from downstream to upstream sampling locations. One field blank will be collected for every twenty samples. ✓ Duplicate samples will be collected at feed water sampling points, whenever feedwater samples are collected.

Chain-of-custody forms should be completed and all samples shipped to ERT's laboratory by overnight delivery on the same day they are collected.

How about effluent?

Sampling points should be flushed for at least five minutes before collecting a sample. Each PAH sample will be collected in four one-liter amber glass bottles, which should be filled and capped in succession. PAH sample bottles should not be rinsed before being filled. The lids of all sample bottles should be taped after they are capped.

All sample bottles should be labeled just before they are used. The date, time, sampler's initials, type of analysis ("PAH"), sample number and unique sample designation should all be recorded on the label.

The GAC treated water samples will have to be collected from two sample taps -- one for each column (see Figure 2-2). This should be done by filling two one-liter bottles from the first column sample tap and then two more bottles from the second (four from each for duplicate samples). No notations distinguishing the two taps should be made on the labels. All four PAH bottles will be extracted and the extracts composited for analysis.

Field blank samples will be prepared by transferring contaminant-free deionized water provided by ERT into sample bottles in a fashion as closely similar to actual sample collection as possible. Field blank sample bottles should be filled, capped and taped in succession with individual bottles open to the atmosphere for an equal time as for actual process samples. Field blanks should be prepared in the area in which GAC treated water samples are collected.

Duplicate samples are obtained by filling eight 1-liter bottles at the sampling point by the procedure described above, splitting these into two groups of four bottles, and assigning a different sample number to each of th resulting four-bottle samples.

All samples should be packed, cooled to a temperature less than 4°C, and shipped on the day they are collected. All sample handling, packaging and shipping should follow ERT's Standard Operating Procedure No. 7510 (Appendix A). One cooler will be used for each day's sampling.

The sampling team must recognize that great care is required to collect samples for part-per-trillion-level PAH analysis that are free from outside contamination. PAH compounds are present in cigarette smoke, engine exhaust and many petroleum derived oils, among other sources. There should be no smoking anywhere in the GAC treatment building on a day on which PAH samples are to be collected until the samples have been collected, sealed and packaged for shipment. Similarly, no vehicles should enter the GAC treatment building and the large access door should stay closed on sampling days. Disposable gloves should be worn when collecting, handling and packaging samples. Sample bottles should remain in closed shipping coolers until they are needed, and should be packaged and sealed for shipment as soon as possible after sampling.

## 2.5 Groundwater Sampling and Water Level Measurements

Ground water samples will be collected and water level measured in accordance with ERT SOP 7130, Ground Water Sample Collection from Monitoring

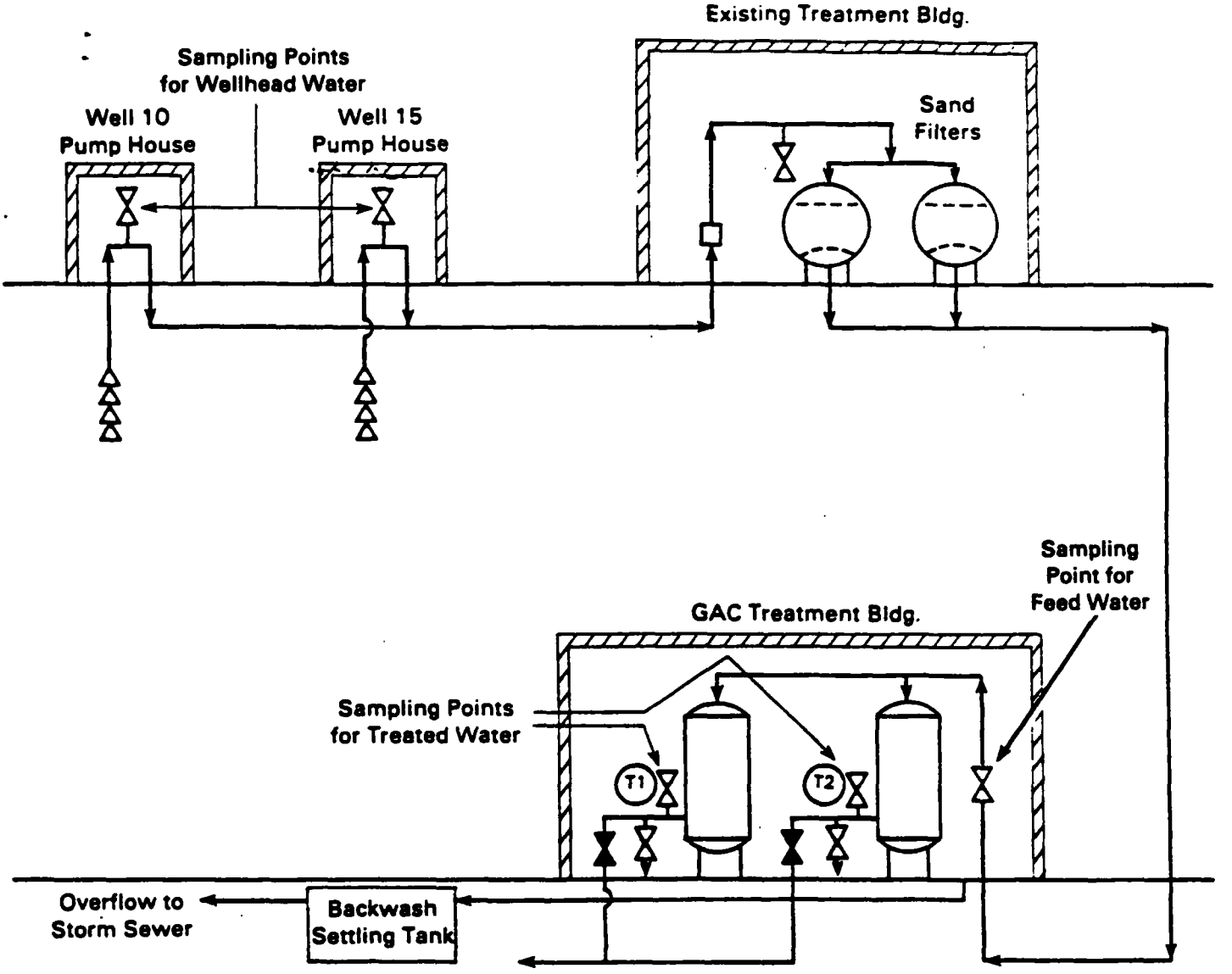



Figure 2-2 Sampling Locations

Wells. The wells involved in the monitoring program include municipal and commercial wells, piezometers and groundwater monitoring wells (see Table 2-1). Sampling procedures to accommodate the dimensions and configuration of each type of well are described below. Further details on well dimensions, configurations and sample acquisition strategies are given in the site management plan. Water level measurements, in accordance with ERT SOP No. 7130 will precede purging and sampling for all monitoring wells and piezometers.

The importance of proper sampling of wells cannot be over-emphasized. Even though the well being sampled may be correctly located and constructed, special precautions must be taken to ensure that the sample taken from that well is representative of the ground water at that location and that the sample is neither altered nor contaminated by the sampling and handling procedure.

2.5.1 Sample Containers (See Table 2-4)

For PAH and Phenolics, 1 liter amber glass bottles should be used. Caps should be fitted with pre-cleaned Teflon liners. Four bottles are required for each PAH sample collected. One bottle is required for phenolics.

Bottles should be prepared as follows:

1. Wash bottles with hot detergent water.

2. Rinse thoroughly with tap water followed by three or more rinses with organic-free water.

3. Rinse with Burdick & Jackson quality redistilled acetone, followed by equivalent quality methylene chloride.

4. Allow to air dry in a contaminant free area.

5. Caps and liners must be washed and rinsed also.

Bottles should be stored and shipped with the Teflon-lined caps securely fastened.

For parameters on the expanded list for the Northern Area of the Drift and Platteville Aquifer, 1-liter amber glass bottles will again be used for the acid and base/neutral extractable organics. Each volatile organics sample will be collected in two forty-milliliter VOA vials. The vials will be prepared in the laboratory before sampling by baking at 110°C for approximately 15 minutes. Samples for metals and ions will be collected in 1-liter polyethylene cubitainers.

bottle blank may be needed!

TABLE 2-4

SAMPLE CONTAINERS, PRESERVATION PROCEDURES, AND MAXIMUM HOLDING TIMES

| Parameter | Containers | Preservation[1] | Maximum Holding Time[2] |
|---|---|---|---|
| Water: | | | |
| PAH (PPT) | Four 1-liter amber glass bottles, Teflon-lined caps | cool, 4°C; protect from light | 7 days (until extraction) |
| PAH (PPB) | Two 1-liter amber glass bottles, Teflon-lined caps | cool, 4°C, protect from light | 7 days (until extraction) |
| Phenolics | One 1-liter amber glass bottle, | cool, 4°C | 7 days (until extraction) |
| Acid, Base/Neutral Extractables | Two 1-liter amber glass bottles, Teflon-lined cap | cool, 4°C (0.008% $Na_2S_2O_3$, if residual Cl is present) | 7 days (until extraction) |
| Volatile Organics | Three 40-ml VOA vials, Teflon septum | cool, 4°C | 14 days |
| Metals | Two 1-liter cubitainers | $HNO_3$ to pH <2 | 6 months |
| $NH_3$, Na, $SO_4$ and Cl | One 1-liter cubitainer | -- | 28 days |

Federal Register Guidelines/Vol.49, No.209/Friday, October 26, 1984/p. 43260.

1. Sample preservation will be performed immediately upon sample collection. For composite samples each aliquot will be preserved at the time of collection, if possible. Each aliquot of the composite, which would require multiple preservatives, will be preserved only by maintaining at 4°C until compositing and sample splitting is completed.

2 Samples will be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still be considered valid.

2.5.2 Sample Collection - Monitoring Wells

Monitoring wells having a riser pipe inside diameter of 4 inches or greater will be purged and sampled using a stainless steel submersible pump with teflon seals and fittings. The pump discharge will be brought to the surface using a polyethylene tube. One tube will be dedicated to each well.

The submersible pump will be decontaminated before use and between sampling points by pumping at least ten gallons of clean water (from GAC plant effluent or uncontaminated municipal well). For each round of sampling with the submersible pump, one field blank will be collected by containerizing a sample of the pump rinsate after at least ten gallons have been pumped.

During the purging of each well, temperature, pH and specific conductance of the purge water will be monitored using a Hydrolab water quality monitor (or equivalent). Readings will be taken once per well volume. Stabilization of these readings will indicate that purging is complete and sampling may commence. All pump discharge not containerized as samples will be directed to a sewer.

Samples are collected by fillling each of the appropriate sample containers in rapid succession. Do not prerinse the containers with sample. Hold the bottle under the sample stream without allowing the mouth of the bottle to come in contact with tubing, pipes, etc. Fill the bottle completely, and securely tighten the cap. Check amber glass bottles and VOA vials for air, if air is visible remove the cap and add more sample. Repeat until no air can be seen in the capped bottle or vial. Be sure all sample labels are completed, as well as sample custody forms and a description of the sampling event recorded in the field notebook.

For every twenty samples (one sample is a complete set of sample containers), a replicate sample should be collected by filling a second set of containers. The replicate sample should be submitted for analysis with the other samples. Field records should clearly identify it as a replicate.

2.5.3 Sample Collection - Piezometers

Piezometers, having a riser pipe inside diameter of less than 4 inches, will be purged and sampled with a peristaltic pump and silicone rubber tubing. Each piezometer will have its own dedicated silicone tube. Pump decontamination will not be required, as the water to be sampled will never come in contact with the pump. Well purging and purge water monitoring for stabilization of temperature, pH and specific conductance will be performed as described in 2.4.2. Sample collection, containerization and replicate sampling will also be as described in 2.4.2.

2.5.4 Sample Collection - Municipal Pumping Wells

Municipal well samples will be acquired from a sampling point as close to the well head as possible. The sampling port will be purged for at least fifteen minutes before a sample is collected. Procedures for sample containerization and replicate sampling will be as described in 2.4.2.

2.6 Sample Preservation, Shipment and Storage

The samples must be iced or refrigerated at 4°C from the time of collection until extraction. PAH's are known to be light sensitive; therefore, samples, extracts and standards will be stored in amber bottles and kept away from prolonged exposure to light. All samples will be extracted within seven days of collection, and analysis completed within forty days following extraction.

Samples should be protected from breakage and shipped in coolers. Ice should be used to maintain a temperature of 4°C. An overnight carrier will be selected to insure delivery at the laboratory within 24-36 hours after collection.

Samples received at the laboratory will be checked for leakage and a notation made regarding sample temperature at time of receipt. All samples should be stored in an organic-free refrigerator at 4°C. Storage refrigerators will be kept locked to prevent unauthorized entry and to satisfy chain-of-custody requirements.

A shipping blank, consisting of four 1-liter amber glass bottles filled with ERT-Laboratory deionized water, will accompany each cooler from the time it is shipped out from the laboratory as a sampling kit until it is retuned with samples for analysis. The shipping blanks will remain in the coolers, capped until their return to the laboratory, at which time they will be analyzed with the samples to detect cross-contamination (if any) that may have occurred during shipment.

2.7 Field Measurement Equipment

All field measurement equipment will be controlled to ensure that measurements obtained are accurate and defensible. Table 2-5 summarizes the routine quality control (QC) checks to be performed on each type of equipment.

In addition, these measurement devices will be issued through a formal equipment tracking system and operated by trained personnel, in accordance with the appropriate SOPs.

is sampling at GAC needed to be addressed too?

TABLE 2-5

FIELD MEASUREMENT EQUIPMENT QUALITY CONTROL

| Device | Calibration | Routine Check Method | Routine Check Frequency | Control Limits |
|---|---|---|---|---|
| pH Meter (Hydrolab) | Standardize in two or more standard buffer solutions | Calibration check-analyze standard buffer solution | 1/10 Samples | To be determined |
| | | Analyze replicates | 1/10 Samples | To be determined |
| Conductivity Meter (Hydrolab) | Standardize using two or more KCL solutions | Calibration check-analyze standard KCL solution | 1/10 Samples | To be determined |
| | | Analyze replicates | 1/10 Samples | To be determined |

control limit needs to be set.

## 3. LABORATORY QUALITY CONTROL

The ERT Analytical Laboratory operates under a formal quality control program governed by ERT's Analytical Laboratory Quality Control Handbook. This section covers quality related activities applicable to the St. Louis Park Groundwater Study from the initiation of sample chain-of-custody to the issuance of validated analytical data. More specific detail of ERT's operation can be found in the handbook.

### 3.1 Chain-of-Custody/and Recordkeeping

When samples are received into the laboratory the sample custodian will verify their integrity as they are unpacked and will explicitly state in the log-in records whether the chain-of-custody seal is intact. The client shall be notified of any discrepancies found and any sample which does not meet the integrity criteria outlined in Chapter 7 of the ERT Analytical Laboratory Quality Control Handbook. If the integrity requirements are met, or when any discrepancies are resolved, ERT assigns the sample a laboratory control number, stores the sample in a refrigerator and enters the pertinent information into the sample log. Once the samples are in the laboratory, a sample usage log is maintained on the LIMS computer to track the transport and use of each sample within the laboratory.

The laboratory will retain a copy of each chain-of-custody record, with the shipper's waybill or air bill attached. After sample log-in, a second copy of the chain-of-custody record will be sent to the Field Coordinator, indicating sample receipt and associated ERT laboratory number. After disposition, the final copy will be sent documenting the disposition method and date.

In addition to sample chain-of-custody, the laboratory will maintain the necessary documentation to reconstruct the entire process of sample preparation through analysis and report generation. This documentation is found in logbooks, data packages and stored on tape.

The logbooks and information they contain are listed below. More thorough descriptions and examples of sample log sheets can be found in ERT's Analytical Quality Control Handbook.

- Chemical Inventory Log - ERT Chemical Inventory control number, compound/reagent name, manufacturer, lot number, grade, date received, expiration date and disposition date.
- Reference Standard Inventory Log - ERT Reference Standard Inventory control number, compound name, manufacturer, lot number, concentration, solvent, date received, expiration and disposition date.



Lab QA

- Super Stock Preparation Log - ERT Super Stock Standard number; neat compound and solvent or carrier name and their pertinent data such as lot number, manufacturer, percent activity, expiration date (if any), weights and volumes taken and balance used; final stock standard concentration, expiration date of standard, storage requirements and location, preparation date and time, preparer's initials, approval signature and date and comments regarding EPA reference standard verification.
- Mixed and/or Dilution Standards Log - ERT Mixed Standard number; pertinent information of Super Stock Standards used such as standard numbers, concentration, preparation date, volume taken, volume diluted to and solvent used (including lot number, manufacturer); mixed and/or dilution standards preparer's initials, date, final concentration of each component, storage, location, approval signature (of supervisor) and date disposed.
- Instrument Maintenance Log - initialed and dated entries pertaining to instrument set-up, routine preventative maintenance, and instrumental malfunction and resolutions.
- Instrument Sample Sequence Log - initialed and dated listing of standards and samples analyzed.
- Instrument Tuning Log - initialed and dated mass intensity listings of daily DFTPP tunes.

The data package contains only data pertinent to the individual project. This package is filed alphabetically by project and date and includes the following records:

- Data Approval Form - a form which lists the contents of the Data Package and routes the data review process.
- Out-of-Control Event Form - a form which describes any out-of-control events which affect the quality of data to be reported and explains the causes and corrective actions taken.
- Sample Receipt Checklist - a checklist describing sample integrity upon receipt into the laboratory.
- Initial Page - a sheet which lists the signatures and initials of all personnel involved in the preparation and review of the Data Package.
- Daily Log Sheet - a log containing daily entries or comments pertaining to any part of sample preparation and/or analysis, which are not described on the other forms such as instrument fluctuations and tuning or where the sample analysis sequence can be found, etc.

- Serial Dilution Sheet - a sheet which is used to describe how dilutions were made from mixed standards to be used as calibration standards or in-house spiking solutions. The following is required: Information about the super stock standards such as parameter, concentration, date prepared, ERT stock standard number, etc. and information about the serial standard preparation such as volume of standard taken, volume diluted to, solvent used, final concentrations, storage location, who prepared it and the date prepared.
- Analytical Results of QA/QC Fortified Samples (Method Spikes) - on this sheet one records pertinent preparation information for spiking samples (GAC treated water) such as volume or weight of sample spiked, concentration of standard used for spiking, and volume of spike used. From this information, one can then calculate the expected concentration of parameter spiked into the method spike sample.

In addition to these forms, a Data Package must contain other pertinent information such as daily instrument calibration, check standard results, chromatographic charts, computer printouts, references to other logbook entries and correspondences. Copies of all GC/MS raw data files are also transferred to magnetic storage media. All data files are maintained in filing cabinets in a secured area for the life of the consent decree.

## 3.2 Quality Control of Analyses

The quality control procedures specific to the analytical method include the determination of method detection limit, the interpretation of the results obtained from method blanks, solvent blanks, surrogates, duplicate samples and method spikes. Laboratory error will be determined based only upon the criteria discussed below for surrogate recovery and method spike recovery.

### Method Detection Limit

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be identified, measured, and reported with 99% confidence that the analyte concentration is greater than zero. This is determined from replicate analyses of a sample of a given matrix containing the analyte near the estimated detection limit.

ERT has determined the method detection limits for the part per trillion PAH analysis of water samples, utilizing GC/MS selected ion monitoring, as per the method described in Appendix B to Part 136 of the Friday, October 26, 1984 Federal Register, Vol. 49, No. 209 - Definition and Procedure for the Determination of the Method Detection Limit - Revision 1.11. Table 3-1 lists the compounds, the mean observed concentration of seven replicates spiked at 5 parts per trillion, the standard deviation, the method detection limit and the lower control limit (defined as 0.64 MDL).

TABLE 3-1

METHOD DETECTION LIMIT STUDY

GC/MS/SIM PART PER TRILLION PAH/HETEROCYCLES IN WATER

| Compound | Mean | Standard Deviation | MDL | Lower Control Limit |
|---|---|---|---|---|
| Naphthalene | 29 | 15 | 47 | 30 |
| → Acenapthylene | 3.1 | 0.53 | 1.7 | 1.1 |
| → Acenapthene | 2.9 | 0.42 | 1.3 | 0.83 |
| Fluorene | 4.3 | 0.28 | 0.88 | 0.56 |
| Phenanthrene | 5.2 | 1.0 | 3.1 | 2.0 |
| Anthracene | 3.8 | 1.1 | 3.4 | 2.2 |
| Fluoranthene | 7.8 | 1.4 | 4.4 | 2.8 |
| Pyrene | 7.7 | 1.3 | 4.1 | 2.6 |
| Benz(a)anthracene | 7.4 | 1.4 | 4.4 | 2.8 |
| Chrysene | 7.6 | 1.4 | 4.4 | 2.8 |
| Benzofluoranthenes | 13 | 3.1 | 9.7 | 6.2 |
| Benzo(a)pyrene | 5.6 | 1.1 | 3.4 | 2.2 |
| Indeno(1,2,3,cd)pyrene | 7.9 | 1.4 | 4.4 | 2.8 |
| Dibenz(a,h)anthracene | 5.5 | 1.1 | 3.4 | 2.2 |
| Dibenzo(g,h,i)perylene | 6.3 | 1.7 | 5.3 | 3.4 |
| Indene | 3.3 | 0.92 | 2.9 | 1.8 |
| Indole | 4.2 | 0.61 | 1.9 | 1.2 |
| 2,3-dihydroindene | 3.7 | 1.1 | 3.4 | 2.2 |
| 2,3-benzofuran | 2.8 | 0.61 | 1.9 | 1.2 |
| Quinoline | 4.5 | 0.61 | 1.9 | 1.2 |
| Benzo(b)thiophene | 4.6 | 0.71 | 2.2 | 1.4 |
| 2-methylnaphthalene | 6.6 | 1.6 | 5.0 | 3.2 |
| 1-methylnaphthalene | 4.9 | 0.98 | 3.1 | 2.0 |
| Biphenyl | 14 | 5.4 | 17 | 11 |
| Carbozole | 5.4 | 0.84 | 2.6 | 1.7 |
| Dibenzofuran | 4.7 | 0.38 | 1.2 | 0.77 |
| Acridine | 3.6 | 0.81 | 2.5 | 1.6 |
| Dibenzothiophene | 4.6 | 2.0 | 6.3 | 4.0 |
| Perylene | 3.5 | 0.52 | 1.6 | 1.0 |
| Benzo(e)pyrene | 4.8 | 0.49 | 1.5 | 0.96 |

All values expressed in part per trillion (ppt)

The study reports should be submitted with the detailed operational procedure of modified method 625.



MDL supposed to be submit with QAPP

Prior to analyzing samples for ppb level PAH using modified EPA Method 625 (as described in Section 4.5). ERT will demonstrate the ability to measure the selected PAH and hetercyclic compounds at 1 ppb by performing a method detection limit study. Within sixty days of submission of this QAPP, a table of results comparable to Table 3-1 will be submitted to the Regional Administrator of the USEPA and the Director of the MPCA for approval of the modified EPA Method 625. No water samples will be analyzed for ppb level PAH prior to receiving the approval.

These calculated method detection limits will be used in sample reporting as follows:

- Concentrations of samples (after blank correction, if applicable) less than the lower control limit of the method detection limit will be reported as not detectable (ND).
- Concentrations of samples (after blank correction, if applicable) greater than the lower control limit of the method detection limit, but less than the method detection limit will be reported as less than the MDL, or BDL (below detection limit).

Method Blank and Solvent Blank

The laboratory will analyze 10% laboratory solvent blanks and 5% method blanks as described in Section 4.0, Analytical Method.

The method blank results associated with the sample batch will be used to correct the observed sample concentrations in that batch as indicated below:

- If the concentration in the blank is less than or equal to half of the method detection limit, samples will not be corrected for the blank.
- If the concentration in the blank is greater than half of the method detection limit and is less than or equal to half the concentration detected in the sample, samples will be corrected for the blank by subtracting the value observed for the compound in the blank from the value observed for the same compound in the sample.
- If the concentration in the blank is greater than half the method detection limit and is greater than half the concentration detected in the sample, correction is not possible and the compound in the sample should be reported as not detected (ND). If this situation occurs, the cause of the high blank must be determined and corrective actions taken (See the ERT Analytical Laboratory Quality Control Handbook, Chapter 8).

The solvent blank is not used to correct sample concentrations, but to help determine the cause of contamination in high blanks.

Check the ERT analytical Lab QC handbook what kind of corrective action specified?

Surrogates

The laboratory will spike all samples and quality control samples with deuterated PAH surrogate compounds. The surrogate compounds will be spiked into the sample prior to extraction and, thus, will measure individual sample matrix effects associated with sample preparation and analysis. They will include naphthalene-$d_8$, fluorene-$d_{10}$ and chrysene-$d_{12}$, (or equivalent compounds) at a sample concentration level of 10 ng/ℓ (ppt) or 20 µg/ℓ (ppb). ERT will calculate the percent recovery of each surrogate for each sample. Prior to beginning work on the project ERT will calculate the 95% confidence limits for each surrogate using historical data. ERT will plot control charts for each surrogate with warning limits at two standard deviations. The control charts will be updated as sample surrogate recoveries are plotted, as a means of observing trends or changes in method precision. Only those surrogate recoveries which meet the acceptance criteria described below will be added to the control charts. Control charts will be used to alert ERT to the need to check method procedures, but failure of a surrogate to fall within the 95% confidence limits of the ongoing control charts does not necessarily invalidate the sample data.

A sample will be invalid for quantitative use in this program only if the recovery of any one or more of the surrogates falls outside the acceptance criteria. The acceptance criteria used for this program are the EPA published acceptance criteria from Method 1625 ("Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act" Federal Register, Friday, October 26, 1984). ERT will take corrective action whenever the surrogate recovery for any one or more surrogates is outside the following acceptance criteria:

| Surrogate | Acceptance Criteria % |
|---|---|
| Naphthalene-d8 | 14-305 |
| Fluorene-d10 | 27-238 |
| Chrysene-d12 | 13-512 |

The following corrective action will be taken when required as stated above:

a) Check calculations to assure there are no errors; check internal standard and surrogate solutions for degradation, contamination, etc.; and check instrument performance.

b) Reanalyze the sample or extract if the steps in part a) fail to reveal a problem. If reanalysis of the extract gives surrogate spike recoveries within the stated limits, then the reanalysis data will be used. In any event, both the original and reanalysis data will be reported.

Should be added prior to the addit. of solvent for extraction

c) If a) or b) fail to correct the problem, the City will be notified by telephone that the sample has been determined unacceptable. ERT will resample within ten (10) working days and reanalyze, providing a report to the City within twenty-eight (28) working days of the resampling.

~~Method~~ Spikes

The laboratory will spike and analyze 5% method spike samples. Following the Contract Laboratory Program rationale, ERT will spike eight representative compounds into deionized water. These compounds and the spiking levels are listed below:

| | PPT | PPB |
|---|---|---|
| Naphthalene | 100 ng/ℓ | 50µg/ℓ |
| Fluorene | 20 | 50 |
| Chrysene | 20 | 50 |
| Benzo(g,h,i)perylene | 20 | 50 |
| Indene | 20 | 50 |
| Quinoline | 20 | 50 |
| Benz(e)pyrene | 20 | 50 |
| 2-methyl naphthalene | 20 | 50 |

Naphthalene is spiked at a higher level in the ppt method, because of the higher method detection limit. The spiking procedure is outlined in Section 4.0, Analytical Method or in Section 8.2 of EPA Method 625.

matrix

ERT will validate the analytical data by utilizing the ~~method~~ spike sample criteria in conjunction with the surrogate recovery criteria. If the criteria for the ~~method~~ spike are met, only samples which do not meet the surrogate recovery criteria in that batch will be considered invalid. If the method spike criteria are not met, the method spike analysis will be repeated. If the subsequent method spike analysis meets the criteria, the data will be considered valid.

The method spike criteria for data validity are as follows:

- The average of the percent recoveries for all eight compounds must fall between 20 and 150 percent.
- Only one compound can be below its required minimum percent recovery. These minimum percent recoveries are:

  1) 10% for chrysene, benzo(g,h,i)perylene, and benz(e)pyrene, and
  2) 20% for all other compounds.

how about matrix spike & spike duplicates?

matrix spikes should be used in conjunction with surrogate recovery in validating data

Duplicates

The laboratory will analyze 10% duplicate samples. Percent difference between duplicates will be calculated for each detected compound. The results will be plotted onto control charts and mean and standard deviation will be calculated.

Additional descriptions of ERT's general laboratory quality control program including control chart construction, interpretation and corrective actions can be found in the ERT Analytical Laboratory Quality Control Handbook.

## 3.3 Corrective Action

The corrective action to be taken when surrogates and method spikes fail to meet their respective criteria are discussed in Section 3.2. This section discusses corrective actions which will be taken in the event that a sample or sample extract is lost or destroyed during shipment, storage or analysis.

Samples

If a sample is broken or lost during shipment, storage or analysis, another sample will be collected and shipped to the laboratory for analysis. The analysis report for the sample batch containing the affected sample will clearly note in the discussion section that a replacement sample was taken.

Sample Extracts

If a sample extract is broken or lost during analysis, another sample will be collected and shipped to the laboratory for analysis. The analysis report for the sample batch containing the affected sample will clearly note in the discussion section that a replacement sample was taken.

Quality Control Samples

If a solvent blank, method blank, or method spike is lost or broken during analysis, a replacement QC sample will be prepared and analyzed. The analysis report will clearly note that a replacement QC sample was analyzed.

If a field blank or shipping blank is lost or broken during shipment, storage, or analysis, no replacement will be analyzed. The analysis report for the sample batch associated with the field or shipping blank will clearly note in the discussion section why the data is unavailable.

## 3.4 Data Review and Reporting

All data will be subjected to a rigorous review process before being reported. All data forms must be dated, signed and completely filled out in ink by the preparer. Notes will be made if information requested is non-applicable for the specific analysis. Each data sheet will be checked, signed, dated and approved by someone other than the preparer.

Out-of-control events or potential out-of-control events are noted on an out-of-control event form. This form is part of the data package and will be completed upon data approval. If no out-of-control events are encountered then this will also be documented. If an out-of-control event does occur during analysis, for instance a surrogate recovery falls outside the expected range, the analyst will describe the event, the investigative and corrective action taken and the cause of the event on this form, and will notify the Quality Control Coordinator (QCC).

After an analyst completes a Data Package, it is given to the Supervisor for review. The Supervisor reviews the entire Data Package for completeness, discrepancies and errors and writes comments, when necessary, on the back of the Data Approval Form. If the supervisor disapproves the Data Package it is given back to the analyst for correction. If it is approved the Supervisor passes it along to the QCC.

The QCC then reviews the Data Package with extra emphasis on the acceptability of quality control data. If the QCC disapproves the Data Package it is rerouted to the Supervisor for corrective action; if the QCC approves it, it is sent to the Laboratory Manager for final approval and report preparation.

Before submission to the client, the final typed report is reviewed by the Program Manager, Laboratory Manager, Supervisors and Quality Control Coordinator for their approval and signatures.

What is included in the data package is not addressed.
- Summary of data only or plus Raw data?

# 4. ANALYTICAL METHODS

## 4.1 Low Level (PPT) Analysis of PAH and Heterocycles

### 4.1.1 Summary

This method has been designed for the analysis of carcinogenic PAH and heterocycles at the part per trillion level (ppt, ng/L) in water. The analysis is carried out by isolation of the target analytes by liquid-liquid extraction of the water sample with an organic solvent. Quantitation of the isolated target analytes is performed by gas chromatography mass spectrometry (GC/MS) in the selected ion monitoring mode (SIM). The compounds listed in Table 4-1 can be quantitatively determined using this analytical method.

Four 1-liter volumes of sample are separated into two 2-liter samples and extracted with methylene chloride. Analysis of the combined and concentrated extract is performed by gas chromatography/mass spectrometry using the selected ion monitoring scanning mode under electron impact ionization conditions.

### 4.1.2 Inteferences

Method interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing hardware that lead to discrete artifacts and/or elevated baselines in the ion current profiles. All of these materials must be routinely demonstrated to be free from interferences under the conditions of the analysis by running laboratory reagent blanks.

Matrix interferences may be caused by contaminants that are coextracted from the sample. The extent of matrix interferences will vary considerably from source to source, depending upon the nature of the environment being sampled.

### 4.1.3 Apparatus

Glassware

Glassware must be scrupulously cleaned. Clean all glassware as soon as possible after use by rinsing with the last solvent used in it. This should be followed by detergent washing with hot water, and rinses with tap water, reagent water, then methanol. It should then be oven dried at 150°C for 30 minutes, and heated in a muffle furnace at 400°C for 15 to 30 minutes. Solvent rinses with methylene chloride may be substituted for the muffle furnace heating. Volumetric glassware should not be heated in a muffle furnace. After drying and cooling, glassware should be sealed and stored in



Analytical Methods

TABLE 4-1

COMPOUNDS AND MS QUANTITATION MASS IONS

| Compound | Quantitation Mass Ion | Confirmation Ion (% Abundance) | Internal Standard Reference |
|---|---|---|---|
| **Polynuclear Aromatic Hydrocarbons (PAH)** | | | |
| Naphthalene | 128 | 102 (20) | 1 |
| Acenaphthylene | 152 | 151 (20) | 1 |
| Acenaphthene | 154 | 153 (90) | 1 |
| Fluorene | 166 | 165 (80) | 2 |
| Phenanthrene | 178 | 176 (20) | 2 |
| Anthracene | 178 | 176 (20) | 2 |
| Fluoranthene | 202 | 200 (20) | 2 |
| Pyrene | 202 | 200 (20) | 2 |
| Benzo(a)anthracene | 228 | 226 (20) | 3 |
| Chrysene | 228 | 226 (20) | 3 |
| Benzofluoranthenes | 252 | 250 (25) | 3 |
| Benzo(a)pyrene | 252 | 250 (25) | 3 |
| Indeno(1,2,3,cd)pyrene | 276 | 274 (20) | 3 |
| Dibenz(a,h)anthracene | 278 | 276 (20) | 3 |
| Benzo(g,h,i)perylene | 276 | 274 (20) | 3 |
| Internal Standards | | | |
| 1) Acenaphthene-d10 | 164 | | - |
| 2) Phenanthrene-d10 | 188 | | - |
| 3) Benz(a)pyrene-d12 | 264 | | - |
| Surrogates | | | |
| 1) Naphthalene-d8 | 136 | | 1 |
| 2) Flourene-d10 | 176 | | 2 |
| 3) Chrysene-d12 | 240 | | 3 |

TABLE 4-1 (Continued)

COMPOUNDS AND MS QUANTITATION MASS IONS

| Compound | Quantitation Mass Ion | Confirmation Ion (% Abundance) | Internal Standard Reference |
|---|---|---|---|
| **Heterocycles and Other PAH** | | | |
| Indene | 116 | 115 (90) | 1 |
| Indole | 117 | 90 (40) | 1 |
| 2,3-dihydroindene | 118 | 117 (50) | 1 |
| 2,3-benzofuran | 118 | 90 (40) | 1 |
| Quinoline | 129 | 102 (30) | 2 |
| Benzo(b)thiophene | 134 | -- | 2 |
| 2-methyl napthalene | 141 | 115 (40) | 2 |
| 1-methyl napthalene | 141 | 115 (40) | 2 |
| Biphenyl | 154 | 153 (30) | 3 |
| Carbazole | 167 | 166 (25) | 3 |
| Dibenzofuran | 168 | 139 (25) | 3 |
| Acridine | 179 | 178 (25) | 3 |
| Dibenzothiophene | 184 | 139 (20) | 3 |
| Perylene | 252 | 250 (30) | 3 |
| Benzo(e)pyrene | 252 | 250 (30) | 3 |
| **Internal Standards** | | | |
| 1) Acenaphthene-d10 | 164 | | - |
| 2) Phenanthrene-d10 | 188 | | - |
| 3) Benz(a)pyrene-d12 | 264 | | - |
| **Surrogates** | | | |
| 1) Naphthalene-d8 | 136 | | 1 |
| 2) Flourene-d10 | 176 | | 2 |
| 3) Chrysene-d12 | 240 | | 3 |

a clean environment to prevent any accumulation of dust or other contaminants. Store it inverted or capped with aluminum foil. The use of high purity reagents and solvents helps to minimize interference problems. Purification of solvents by distillation in all-glass systems may be required.

a) Separatory funnel - 2000 mL, with Teflon stopcock.

b) Concentrator tube, Kuderna-Danish - 10 mL, graduated (Kontes K-570050-1025 or equivalent). Calibration must be checked at the volumes employed in the test. Ground-glass stopper is used to prevent evaporation of extracts.

c) Snyder column, Kuderna-Danish - Three-ball macro (Kontes K-503000-0121 or equivalent).

d) Evaporative flask, Kuderna-Danish - 500 mL (Kontes K-570001-0500 or equivalent). Attach to concentrator tube with springs.

e) Snyder column, Kuderna-Danish - two-ball micro (Kontes K-569001-0219 or equivalent).

f) Micro reaction vessels, 2.0 mL (Supelco 3-3295).

Gas Chromatograph

The analytical system is complete with a temperature programmable gas chromatograph and all required accessories including syringes, analytical columns, and gases. The injection port is designed for on-column injection when using packed columns and for splitless injection when using capillary columns.

Column

A J&W 15-meter fused silica capillary column coated with DB-5 bonded phase, or equivalent.

Mass Spectrometer

A mass spectrometer operating at 70 ev (nominal) electron energy in the electron impact ionization mode and producing a mass spectrum which meets all the ion abundance criteria when 50 ng of decafluorotriphenyl phosphine (DFTPP; bis(perfluorophenyl) phenyl phosphine) is injected through the GC inlet. The GC capillary column is fed directly into the ion source of the mass spectrometer.

how the extraction is done with 2l funnel?

A computer system interfaced to the mass spectrometer allows the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer has software that allows searching any GC/MS data file for ions of a specific mass and plotting such ion abundances versus time or scan number. The computer allows acquisition at pre-selected mass windows for selected ion monitoring.

Reagents

a) Reagent water - Reagent water is defined as a water in which an interferent is not observed at the method detection limit of each parameter of interest.

b) Solvents - Acetone, methanol, methylene chloride, benzene, cyclohexane - Burdick & Jackson, distilled in glass, or equivalent.

c) Sodium sulfate - (ACS) Granular, anhydrous. Purify by heating at 400°C for 4 hrs. in a shallow tray.

d) Surrogate Spiking Solution - A solution containing 10 ng/mL of each of naphthalene-$d_8$, fluorene-$d_{10}$, and chrysene-$d_{12}$ (or equivalent weight deuterated PAH) is prepared by weighing appropriate aliquots of the purified crystals into a volumetric flask and dilution to volume with methanol or acetone.

e) Internal Standard Solutions - A solution containing ca. 200 ng/mL of each internal standard is prepared by weighing an appropriate aliquot of each purified crystal into a volumetric flask and diluting to volume with methylene chloride. The internal standard compounds are acenaphthene-d10, phenanthrene-d10, and benzo(a)pyrene-d12, or equivalent weight deuterated PAH, not used as a surrogate.

f) Matrix Recovery Standard Spiking Solution - A solution containing the following compounds at the listed concentrations is prepared by weighing an appropriate aliquot of each purified crystal into a volumetric flask and diluting to volume with methanol or acetone.

f) is this the spike soln used for method spike.?

| Compound | Concentration (ng/mL) |
|---|---|
| Naphthalene | 100 |
| Fluorene | 20 |
| Chrysene | 20 |
| Benzo(g,h,i) perylene | 20 |
| Indene | 20 |
| Quinoline | 20 |
| Benz(e)pyrene | 20 |
| 2-methylnaphthalene | 20 |

4.1.4 Extraction

Samples

Samples are extracted at pH >12. Each 4-liter sample is separated into two 2-liter aliquots in two 2-liter separatory funnels. Each 2-liter aliquot is spiked in the separatory funnel with the surrogate spiking solution. A 2.00 mL volume of mixed surrogate spiking standard is added to each 2-liter separatory funnel, to give an approximate concentration of 10 ng/L (10 ppt) of each surrogate. Each aliquot is then extracted three times (80 mL/80 mL/80 mL) with methylene chloride. The three methylene chloride extracts are passed through an anhydrous sodium sulfate drying column, and combined in a Kuderna-Danish evaporative concentrator.

Concentrate the extract to ca. 0.5 mL and transfer to a 2.0 mL microreaction vessel containing 0.5 mL (500 ul) of benzene. The methylene chloride is evaporated using a nitrogen stream. The evaporative concentrator tube is successively rinsed with methylene chloride, the rinsings added to the reaction vessel and the methylene chloride again evaporated. Continue this process until at least five (5) 1 mL rinsings of the tube have occurred. Evaporate the final methylene chloride, leaving the 500 ul of benzene. All microreaction vessels should be permanently marked at the 500 µl level and additional benzene added, when necessary, to insure a final 500 µl extract volume. Cap with a Teflon fitted septum cap and store the extract at 4°C prior to GC/MS analysis.

Method Blank

For a minimum of 5% of the analyses performed, prepare a method blank by treating a 4-L sample of laboratory reagent water exactly as described above.

Solvent Blank

For a minimum of 10% of the analyses performed, prepare a solvent blank by introducing methylene chloride into two clean 2-liter separatory funnels (80 mℓ/80 mℓ/80 mℓ). Combine the methylene chloride extracts and continue the concentration exactly as described above.

Matrix Recovery Sample

For a minimum of 5% of the analyses performed, prepare a matrix recovery sample by spiking 2.00 mL of the matrix recovery standard spiking solution into two 2-L volumes of laboratory reagent water. Extract the fortified sample exactly as described above for samples. At this level of spiking, the following compounds will be introduced into the 4-L sample at the following concentrations:

| Compound | Concentration (ng/mL) |
|---|---|
| Naphthalene | 100 |
| Fluorene | 20 |
| Chrysene | 20 |
| Benzo(g,h,i) perylene | 20 |
| Indene | 20 |
| Quinoline | 20 |
| Benz(e)pyrene | 20 |
| 2-methylnaphthalene | 20 |

Conc. of the spike mixture or the actual conc. in the 4 liter water.

Duplicate Sample

For a minimum of 10% of the samples analyzed a duplicate sample will be taken at sampling and a duplicate analysis will be performed. This will be carried out to insure that an estimate of precision will be available.

4.1.5 GC/MS Calibration

Prior to use of this method a five-point response factor calibration curve must be established showing the linear range of the analysis. For every 12 hours of GC/MS analysis, the mass spectrometer response for each PAH or heterocycle relative to the internal standard is determined, as described in the Calculations Section, using daily check standards at concentrations of 40 ng/mL. Daily response factors for each compound must be compared to the initial calibration curve. If the daily response factors are within ±35 percent of the corresponding calibration curve value the analysis may proceed. If, for any analyte, the daily response factor is not

project samples should be used instead of reagent water

within ±35 percent of the corresponding calibration curve value, a five-point calibration curve must be repeated for that compound prior to the analysis of samples.

Chromatographic peak location criteria will be established using relative retention time. An initial determination of retention times for each PAH or heterocycle relative to its respective internal standard (Table 4-1) will be made using five-point calibration standards. Average relative retention times and standard deviations will be calculated and 95 percent confidence limits established. Relative retention times of daily calibration standards must be within these 95 percent confidence limits for each PAH or heterocyclic compound. In addition, sample component relative retention times must be within ±0.1 relative retention time units of the standard component relative retention time.

4.1.6 Daily GC/MS Performance Tests

At the beginning of each 12 hour shift that analyses are to be performed, the GC/MS system must be checked to see that acceptable performance criteria are achieved for DFTPP. This DFTPP performance test requires the following instrumental parameters:

Electron Energy 70 volts (nominal)
Mass Range - 35 to 450 amu
Scan Time - 1.0 sec.

At the beginning of each 12 hour shift, inject 2 µL (50 ng) of DFTPP standard solution. Obtain a background corrected mass spectrum of DFTPP and check that all the key ion criteria in Table 4-2 are achieved. If all the criteria are not achieved, the analyst must retune the mass spectrometer and repeat the test until all criteria are achieved.

4.1.7 Gas Chromatography/Mass Spectrometry Analysis

Just prior to analysis a 125 µl aliquot of internal standard solution is transferred to the sample vial using a 250 µL syringe, giving a final internal standard concentration of ca. 40 ng/mL in the extract. Representative aliquots are injected into the capillary column of the gas chromatograph using the following conditions:

Injector Temp - 290°C
Transfer Line Temp - 310°C
Initial Oven Temp - 35°C
Initial Hold Time - 2 min.
Ramp Rate - 10°C/min.
Final Temperature - 310°C

TABLE 4-2

DFTPP ION ABUNDANCE CRITERIA

| Mass | Ion Abundance Criteria |
|---|---|
| 51 | 30 to 60 percent of mass 198 |
| 68 | less than 2 percent of mass 69 |
| 70 | less than 2 percent of mass 69 |
| 127 | 40 to 60 percent of mass 198 |
| 197 | less than 1 percent of mass 198 |
| 198 | base peak, 100 percent |
| 199 | 5 to 9 percent of mass 198 |
| 275 | 10 to 30 percent of mass 198 |
| 365 | greater than 1 percent of mass 198 |
| 441 | present but less than mass 443 |
| 442 | greater than 40 percent of mass 198 |
| 443 | 17 to 23 percent of mass 442 |

The effluent from the GC capillary column is fed directly into the ion source of the mass spectrometer. The MS is operated in the selected ion monitoring (SIM) mode using appropriate windows to include the quantitation and confirmation masses of each PAH or heterocycle as shown in Table 4-1. The time programmed SIM acquisition windows are listed in Table 4-3. Each SIM sequence is acquired at a total scan speed of 1.1 seconds per scan. Typical retention behavior of the combined PAH and heterocycle analytes and corresponding SIM sequences are shown in Table 4-4. For all compounds detected at a concentration above the MDL, a check is made to insure the confirmation ion is present.

Calculations

The following formula is used to calculate the response factors of the internal standard to each of the calibration standards.

$RF = (A_s C_{is})/(A_{is} C_s)$

where:

$A_s$ = Area of the characteristic ion for the parameter to be measured.
$A_{is}$ = Area of the characteristic ion for the internal standard.
$C_{is}$ = Concentration of the internal standard, (ng/mL).
$C_s$ = Concentration of the parameter to be measured, (ng/mL).

Based on these response factors, sample extract concentration for each PAH is calculated using the following formula.

$$\text{Concentration, ng/mL} = \frac{(A_s)(I_s)}{(A_{is})(RF)}$$

where:

$A_s$ = Area of the characteristic ion for the parameter to be measured.
$A_{is}$ = Area of the characteristic ion for the internal standard.
$I_s$ = Amount of internal standard added to each extract (ng/mL).

The actual sample concentration (C) for each compound is calculated by the following formula:

TABLE 4-3

SELECTED ION MONITORING (SIM) SEQUENCE FOR PAH AND HETEROCYCLES

| Sequence # | M/Z Scanned | Scan # Range | Start Time (Min) |
|---|---|---|---|
| 1 | 90, 115, 116, 117, 118 | 300-499 | 5.50 |
| 2 | 102, 128, 129, 134, 136 | 500-599 | 9.17 |
| 3 | 90, 115, 117, 141, 153, 154 | 600-719 | 11.00 |
| 4 | 139, 151, 152, 153, 154, 164, 165, 166, 168, 176 | 720-899 | 13.20 |
| 5 | 139, 166, 167, 176, 178, 179, 184, 188 | 900-1049 | 16.50 |
| 6 | 200, 202, 212 | 1050-1249 | 19.25 |
| 7 | 226, 228, 240 | 1250-1399 | 22.92 |
| 8 | 250, 252, 264 | 1400-1649 | 25.67 |
| 9 | 274, 276, 278 | 1650-1850 | 30.25 |

TABLE 4-4

GC RETENTION BEHAVIOR FOR PAH AND HETEROCYCLES

| Compound | M/Z | Retention Scan Number | SIM Sequence # |
|---|---|---|---|
| 2,3-benzofuran | 118 | 383 | 1 |
| 2,3-dihydroindene | 118 | 420 | 1 |
| Indene | 116 | 429 | 1 |
| Napthalene-d8 (Surr.) | 136 | 548 | 2 |
| Napthalene | 128 | 551 | 2 |
| Benzo(b)thiophene | 134 | 557 | 2 |
| Quinoline | 129 | 593 | 2 |
| Indole | 117 | 635 | 3 |
| 2-methylnapthalene | 141 | 640 | 3 |
| 1-methylnapthalene | 141 | 653 | 3 |
| Biphenyl | 154 | 703 | 3 |
| Acenaphthylene | 152 | 756 | 4 |
| Acenaphthene-d10 (IS-1) | 164 | 776 | 4 |
| Acenaphthene | 154 | 781 | 4 |
| Dibenzofuran | 168 | 802 | 4 |
| Fluorene-d10 (Surr.) | 176 | 843 | 4 |
| Fluorene | 166 | 848 | 4 |
| Dibenzothiophene | 184 | 956 | 5 |
| Phenanthrene-d10 (IS-2) | 188 | 970 | 5 |
| Phenanthrene | 178 | 974 | 5 |
| Anthracene | 178 | 980 | 5 |
| Acridine | 179 | 985 | 5 |
| Carbazole | 167 | 1004 | 5 |
| Fluoranthene | 202 | 1134 | 6 |
| Pyrene | 202 | 1162 | 6 |
| Benz(a)anthracene | 228 | 1333 | 7 |
| Chrysene-d12 (Surr.) | 240 | 1335 | 7 |
| Chrysene | 228 | 1339 | 7 |
| Benzofluoranthenes | 252 | 1496 | 8 |
| Benz(e)pyrene | 252 | 1536 | 8 |
| Benz(a)pyrene-d12 (IS-3) | 264 | 1539 | 8 |
| Benz(a)pyrene | 252 | 1543 | 8 |
| Perylene | 252 | 1546 | 8 |
| Indeno (1,2,3-cd)pyrene | 276 | 1713 | 9 |
| Dibenz(a,h)Anthracene | 278 | 1718 | 9 |
| Benzo(g,h,i)Perylene | 276 | 1750 | 9 |

$$C, \text{ ng/L (ppt)} = \text{Extract Concentration} \times \frac{V_E}{V_S} ,$$

where

$V_E$ = The final extract volume (mL), and
$V_S$ = The original volume of sample extracted (L).

4.2 Extended Analyses for Carcinogenic PAH in GAC Plant

To satisfy the requirements of the RAP Section 4.3.4, ERT will analyze one sample per year of the GAC treated water for the additional carcinogenic compounds shown on Table 4-5 and search for additional compounds which may be present. ERT will first analyze the sample according to Section 4.1 of this QAPP. A calibration standard containing the compounds shown on Table 4-5 will be prepared and used to establish a five point calibration curve. All procedures outlined in Section 4.1 for instrument calibration will be followed.

The sample extract will be prepared and analyzed as outlined in Section 4.1 generating quantitative results for the compounds being regularly measured. A second injection will be made with a selective ion monitoring program using the quantitation masses shown in Table 4-5. This will allow the extended analysis compounds to be quantitated at a ca. 2ppt detection limit.

2 injection or 2 analysis per sample.

Following the quantative analyses of the regular and extended analysis compounds, the extract will be reduced to a 50 ul final volume. An aliquot will be analyzed using full-scan GC/MS. Any peaks having a signal to noise ratio of 5 or larger will be identified, if possible, using the EPA/NIH mass spectral library. Compounds so identified will be quantitated using the nearest internal standard and a response factor of 1.0, to a detection limit of ca. 5 ppt.

4.3 Extended Analyses for Phenolics in GAC Plant

To satisfy the requirements of the RAP Section 4.3.4, ERT will analyze one sample per year of GAC treated water for the acid extractable compounds shown on Table 4-6. These compounds will be analyzed according to sections applicable to acid extractables in EPA Method 625 ("Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act". Federal Register, Friday, October 26, 1984).

① Is the Sample extract used for PAH and extended anlysis to be used for phenol analysis?
② If not, how the ? Table will be prepared

Will the conc. be high enough for GC/MS. Req 10 fold increase in conc.?

is different from GAC QAPP.

is this to be done after the performance of GAC is demonstrate to be satisfactory — other QAPP?

TABLE 4-5

EXTENDED ANALYSIS CARCINOGENIC PAH

| Compound | Quantitation Mass |
|---|---|
| benzo(c)phenanthrene | 226 |
| dibenz(a,c)anthracene | 278 |
| dibenz(a,e)pyrene | 276 |
| dibenz(a,h)pyrene | 276 |
| dibenz(a,i)pyrene | 276 |
| 7,12-dimethylbenz(a)anthracene | 256 |
| 3-methylcholanthrene | 268 |

TABLE 4-6

EXTENDED ANALYSES ACID EXTRACTABLES

4-chloro-3-methylphenol

2-chlorophenol

2,4-dichlorophenol

2,4-dimethylphenol

2,4-dinitrophenol

2-methyl-4,6-dinitrophenol

2-nitrophenol

4-nitorphenol

Pentachlorophenol

Phenol

2,4,6-trichlorophenol

## 4.4 Expanded Analyses

In accordance with RAP, Section 9.3.3, the Regional Administrator, the Director, or the Commissioner may request expanded analyses in conjunction with the Northern Area Remedial Investigation. The list of possible analyses are shown on Table 4-7. Organic analyses and metals analyses will be performed at ERT's Concord laboratory facility. The inorganic analyses will be performed at ERT's Houston laboratory facility.

The analytical methods to be used for each analyte are also shown on Table 4-7.

## 4.5 Non-Criteria PAH Analyses

As discussed in the Site Management Plan, selected water samples will be analyzed for PAH at ppb levels (1 ppb detection limit). A 2 liter water sample will be collected and extracted according to EPA Method 625. ERT will use naphthalene-d8, chrysene-d12 and fluorene-d10 at a sample concentration of 20 ug/l (ppb) as surrogates. The solvent extract will be reduced to a final volume of 0.1 ml. An aliquot of the extract will be analyzed by GC/MS as required in EPA Method 625, with the following modifications:

- A three point calibration curve will be prepared for only the compounds listed in Table 4-1 of the QAPP.
- Three internal standards will be used: Acenaphthene-d10, Phenanthrene-d10, and Benz(a)pyrene-d12.
- Matrix spikes will be prepared using the following representative compounds at a sample concentration of 50 ug/l (ppb):

  Naphthalene
  Fluorene
  Chrysene
  Benzo(ghi)perylene
  Indene
  Quinoline
  Benzo(e)pyrene
  2-methylnaphthalene

TABLE 4-7

EXPANDED ANALYSES ANALYTE LIST AND

METHOD REFERENCE

| Analytes | Method Reference |
|---|---|
| Volatile Organics | EPA 624[1] |
| Acid, Base/Neutral Extractable Organics | EPA 625[1] |
| Priority Pollutant Metals | EPA 200.7, 204.2, 206.2, 245.1, 270.2, 279.2[2] |
| Ammonia | EPA 350[2] ? 350.1, 350.2 or 350.3 |
| Chloride | EPA 325[2] ? 325.1, 325.2 or 325.3 |
| Sodium | EPA 200.7[2] |
| Sulfate | EPA 375[2] ? .1 ~ .4. which one? |

1 "Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act" Federal Register, Friday, October 26, 1984.

2 "Methods for Chemical Analysis of Water and Wastes" EPA-600/4-79-020, March 1979.

out of date document

Revised march, 1983

# 5. REPORTING

## 5.1 Summary

ERT will provide, for each sample analyzed, six (6) copies of the five (5) QA/QC data reports - the Method Detection Limit Report, the Analytical Results Report, the Surrogate Recovery Report, the Sampling Report, and the Method Spike Recovery Report. These reports will be submitted to the City within 28 working days of sample collection. The following discussion describes in detail the contents of these deliverables. In addition, the reporting requirements for samples found to exceed Advisory Levels or Drinking Water Criterion are discussed.

matrix

## 5.2 Report Descriptions

### 5.2.1 Method Detection Limit Report

The Method Detection Limit Report will consist of a tabulation of method detection limits (MDL) and lower confidence limits (LCL) for each compound analyzed. These concentration limits will be utilized in completing the Analytical Results Report (5.2.3) for all samples analyzed. An example of this report is included as Figure 5-1.

### 5.2.2 Sampling Report

The Sampling report will contain the following information associated with each sample and sample analysis:

1) Field Identification Designation
2) ERT Laboratory Sample Number
3) Field Logbook/Page Number
4) Date of Collection
5) Date Received at ERT
6) Date Extracted
7) Date Analyzed
8) GC/MS File #
9) GC/MS Tape #
10) Corresponding DFTPP File #
11) Corresponding Matrix Spike Sample #
12) Corresponding Method Blank Sample #
13) Corresponding Solvent Blank Sample #
14) Corresponding GC/MS Calibration Standard File #
15) Description of any problems encountered

matrix spike recovery should be used.

Reporting

FIGURE 5-1

ERT ANALYTICAL LABORATORY METHOD DETECTION LIMITS

POLYAROMATIC HYDROCARBONS

| Compound | Method Detection Limit (MDL) | Lower Control Limit (LCL) |
|---|---|---|
| Naphthalene | 47 | 30 |
| Acenapthylene | 1.7 | 1.1 |
| Acenapthene | 1.3 | 0.83 |
| Fluorene | 0.88 | 0.56 |
| Phenanthrene | 3.1 | 2.0 |
| Anthracene | 3.4 | 2.2 |
| Fluoranthene | 4.4 | 2.8 |
| Pyrene | 4.1 | 2 |
| Benz(a)anthracene | 4.4 | |
| Chrysene | | 2.8 |
| Benzofluoranthenes | 9. | 6.2 |
| Benzo(a)pyrene | 3.4 | 2.2 |
| Indeno(1,2,3,cd)pyrene | 4.4 | 2.8 |
| Dibenz(a,h)anthracene | 3.4 | 2.2 |
| Dibenzo(g,h,i)perylene | 5.3 | 3.4 |
| Indene | 2.9 | 1.8 |
| Indole | 1.9 | 1.2 |
| 2,3-dihydroindene | 3.4 | 2.2 |
| 2,3-benzofuran | 1.9 | 1.2 |
| Quinoline | 1.9 | 1.2 |
| Benzo(b)thiophene | 2.2 | 1.4 |
| 2-methylnaphthalene | 5.0 | 3.2 |
| 1-methylnaphthalene | 3.1 | 2.0 |
| Biphenyl | 17 | 11 |
| Carbozole | 2.6 | 1.7 |
| Dibenzofuran | 1.2 | 0.77 |
| Acridine | 2.5 | 1.6 |
| Dibenzothiophene | 6.3 | 4.0 |
| Perylene | 1.6 | 1.0 |
| Benzo(e)pyrene | 1.5 | 0.96 |



need a real one.

All values expressed in part per trillion (ppt)

5.2.3 Analytical Results Report

Each analytical results report will contain the following:

1) Field Identification Designation
2) ERT Laboratory Sample Number
3) Analytical Results (ppt or ppb), in terms of a) individual PAH identification and quantitation b) Total Carcinogenic PAH c) Total Other PAH and d) Total PAH

The analytical results report will be validated and signed by the Laboratory Manager.

List of Carcinogenic PAH and Other PAH

The analytical method will provide for identification and quantitation of two groups of target compounds - the Carcinogenic PAH and the other PAH group. Listed in Table 5-1 are the two groups of target compounds. Analytical results will be reported for individual compounds, with the exception of the three benzofluoranthene isomers (b,j, and k). Due to the difficulty in maintaining chromatographic separation of this isomeric series, a total benzofluoranthenes analytical result will be reported. This benzoflouranthenes quantitative result will be utilized in the calculation of total carcinogenic PAH.

Analytical Results Reporting Protocol

The quantitative results for any of the identified target compounds will be reported in one of three possible ways. Concentrations of analytes equal to or greater than the method detection limit (MDL) will be assigned a numerical concentration value reported to two (2) significant figures (i.e. 52 ng/L). Concentrations of analytes identified as present at a level less than the MDL but equal to or greater than the lower confidence limit (LCL) of the 95% confidence interval of the MDL are reported as less than the MDL (<MDL, i.e. <3.0 ng/L). Concentrations of target analytes less than the LCL (95% confidence interval) of the MDL are reported as not detectable (i.e. ND). In all cases, the quantitative results will be corrected for levels observed in the method blank, as described in Section 3.3. An example of this report is included as Figure 5-2.

TABLE 5-1

STANDARD PAH AND OTHER PAH COMPOUNDS

FOR IDENTIFICATION AND QUANTITATION

a. Carcinogenic PAH

| Compound | Chemical Abstract Service Registry No. |
|---|---|
| benzo(a)anthracene | ( 56-55-3) |
| benzo(b)fluoranthene | (205-99-2) |
| benzo(j)fluoranthene | (205-82-3) |
| benzo(k)fluoranthene | (207-08-9) |
| benzo(ghi)perylene | (191-24-2) |
| benzo(a)pyrene | ( 50-32-8) |
| chrysene | (218-01-9) |
| dibenz(a,h)anthracene | ( 53-70-3) |
| indeno(1,2,3-cd)pyrene | (193-39-5) |
| quinoline | ( 91-22-5) |

b. Other PAH

| Compound | Chemical Abstract Service Registry No. |
|---|---|
| acenaphthene | ( 83-32-9) |
| acenaphthylene | (208-96-8) |
| acridine | (260-94-6) |
| anthracene | (120-12-7) |
| 2,3-benzofuran | (271-98-6) |
| benzo(e)pyrene | (192-97-2) |
| benzo(b)thiophene | ( 95-15-8) |
| biphenyl | ( 92-15-8) |
| carbazole | ( 86-74-8) |
| dibenzofuran | (132-64-9) |
| dibenzothiophene | (132-65-0) |
| 2,3-dihydroindene | (496-11-7) |
| fluoranthene | (206-44-0) |
| fluorene | ( 86-73-7) |
| indene | ( 95-13-6) |
| indole | (120-72-9) |
| 1-methylnaphthalene | ( 90-12-0) |
| 2-methylnaphthalene | ( 91-57-6) |
| naphthalene | ( 1-20-3) |
| perylene | (198-55-0) |
| phenanthrene | ( 85-01-08) |
| pyrene | (129-00-0) |

FIGURE 5-2

ERT ANALYTICAL LABORATORY

SUMMARY OF ANALYTICAL RESULTS

POLYAROMATIC HYDROCARBONS

Field ID: W-02 ERT No: 37015



CARCINOGENIC PAHs

| Parameters | Analytical Result (ng/l) |
|---|---|
| Quinoline | ND |
| Benzo(a)anthracene | ND |
| Chrysene | ND |
| Benzofluoranthenes | ND |
| Benzo(a)pyrene | ND |
| Indeno(1,2,3-CD)pyrene | ND |
| Dibenz(a,h)anthracene | ND |
| Benzo(g,h,i)perylene | ND |
| Total Carcinogenic PAH | ND |

OTHER PAHs

| | |
|---|---|
| 2,3-benzofuran | ND |
| 2,3-dihydroindene | 7.7 |
| indene | ND |
| Naphthalene | ND |
| Benzo(b)thiophene | ND |
| Indole | ND |
| 2-methylnaththalene | ND |
| 1-methylnaphthalene | ND |
| Biphenyl | ND |
| Acenaphthylene | 7.5 |
| Acenaphthene | 11 |
| Dibenzofuran | <1,2 |
| Fluorene | 4.5 |
| Dibenzothiophene | ND |
| Anthracene | <3.4 |
| Acridine | ND |
| Carbazole | ND |
| Fluoranthene | ND |
| Pyrene | 4.5 |
| Benzo(e)pyrene | ND |
| Perylene | ND |
| Total Other PAH: | 35 |
| Total PAHs: | 35 |

ND = Concentration <LCL of MDL
<MDL = Concentration >LCL but <MDL

5.2.4 Surrogate Recovery Report

Each surrogate recovery report will contain the following:

1) Field Identification Designation
2) ERT Laboratory Sample Number
3) Spiking concentration for each of the three deuterium labelled surrogate compounds (naphthalene-$d_8$, fluorene-$d_{10}$, chrysene-$d_{12}$)
4) Percent recovery result for each of the three surrogate compounds.

An example of this report is included as Figure 5-3.

5.2.5 Method Spike Recovery Report

Each method spike recovery report will contain the following:

1) Field identification designation
2) ERT laboratory sample number
3) Spiking concentrations for each of the eight compounds selected (naphthalene, fluorene, chrysene, benzo(g,h,i) perylene, indene, quinoline, benz(e)pyrene, and 2-methylnaphthalene).
4) Percent recovery results for all the method spike compounds.
5) Average percent recovery for the group of eight compounds spiked.

An example of this report is included as Figure 5-4.

5.2.6 Reporting Requirements for Samples Exceeding Advisory Levels or Drinking Water Criterion

For active drinking water wells, ERT will notify the City of St. Louis Park by telephone, within 24 hours of completing an analysis, whenever a sample analysis is shown to exceed the following Advisory Levels or Drinking Water Criterion:

| Parameter | Advisory Level | Drinking Water Criterion |
|---|---|---|
| Sum of Benzo(a)pyrene and Dibenz(a,h)anthracene | 3.0 ng/L | 5.6 ng/L |
| Total Carcinogenic PAH | 15 ng/L | 28 ng/L |
| Total Other PAH | 175 ng/L | 280 ng/L |

Should be matrix spike instead of method spike.

FIGURE 5-3

KRT ANALYTICAL LABORATORY SUMMARY OF ANALYTICAL RESULTS

SURROGATE RECOVERY REPORT

POLYAROMATIC HYDROCARBONS

Field Id: W-02 KRT No: 37015



| Surrogate | Spike Level (ng/l) | Recovery % |
|---|---|---|
| Naphthalene - D8 | 9.9 | 35 |
| Fluorene - D10 | 9.5 | 103 |
| Chrysene - D12 | 9.8 | 80 |

FIGURE 5-4

ERT ANALYTICAL RESULTS QUALITY CONTROL CHECK

SAMPLES POLYAROMATIC HYDROCARBONS

Field Id: MS-02 ERT No: 37018

| Parameters | Spike Level (ng/l) | Recovery % |
|---|---|---|
| Naphthalene | [illegible] | 49 |
| Fluorene | [illegible] | 43 |
| Chrysene | [illegible] | 60 |
| Benzo(g,h,i)perylene | 22.4 | 9 |
| Indene | 24.6 | 28 |
| Quinoline | 23.5 | 52 |
| Benzo(e)pyrene | 20.4 | 12 |
| 2-methylnaphthalene | 21.2 | 50 |
| Average % Recovery: | | 38 |

A written report shall be submitted by ERT within 2 working days following the original telephone notification. In the event it is determined by the City that the analytical results were achieved due to improper procedures or practices, ERT will note this finding and proceed with retesting as directed by the City. Subsequent retesting will be completed with a written report submitted to the City within eighteen (18) days of receipt of notice to proceed (issued either verbal or written). Furthermore, in the event the City determines that all procedures of the analysis were proper and that a defined level had been exceeded, ERT will complete the necessary retest, including submittal of the written report, within eighteen (18) days of receipt of notice to proceed (issued either verbal or written).

The City shall submit the written report to the agencies within 21 days of the retest.

# APPENDIX A

## STANDARD OPERATING PROCEDURES

ERT STANDARD OPERATING PROCEDURE

Number: 7130 Date of Issue: March 12, 1984

Title: Ground-Water Sample Collection from Monitoring Wells

| Organizational Acceptance | Authorization | Date |
|---|---|---|
| Originator | Christopher Carlo | [illegible] |
| Department Manager | Austin Lazarus | 3/12/84 |
| Divisional Manager | | |
| Group Quality Assurance Officer | | |
| Other | | |

| Revisions | Changes | Authorization | Date |
|---|---|---|---|

## 1.0 Applicability

This Standard Operating Procedure (SOP) is concerned with the collection of valid and representative samples from ground-water monitoring wells. The scope of this document is limited to field operations and protocols applicable during ground-water sample collection.

## 2.0 Responsibilities

The site coordinator or his delegate will have the responsibility to oversee and ensure that all ground-water sampling is performed in accordance with the project-specific sampling program and this SOP. In addition, the site coordinator must ensure that all field workers are fully apprised of this SOP. The field team is responsible for proper sample handling as specified in SOP 7510, Handling and Storage of Samples.

## 3.0 Supporting Materials

The list below identifies the types of equipment which may be used for a range of ground water-sampling applications. From this list, a project-specific equipment list will be selected based upon project objectives, the depth to ground-water, purge volumes, analytical parameters and well construction. The types of sampling equipment are as follows:

- Purging/Sample Collection

  Bailers
  Centrifugal Pump
  Submersible Pump
  Peristaltic Pump

- Sample Preparation/Field Measurement

  pH Meter
  Specific Conductance Meter
  Filtration Apparatus
  Water-Level Measurement Equipment

Additional equipment to support sample collection and provide baseline worker safety will be required to some extent for each sampling task. The additional materials are separated into two primary groups: general equipment which is reusable for several samplings, and materials which are expendable.

- General

  Project-specific sampling program
  Goggles or equivalent eye protection
  Full- or half-mask respirators
  Deionized-water dispenser bottle
  Methanol-dispenser bottle

  Field data sheets and/or log book
  Preservation solutions
  Sample containers
  Buckets and intermediate containers
  Coolers
  First-Aid kit

- Expendable Materials

  Bailer Cord
  Respirator Cartridges
  Gloves
  Water Filters
  Chemical-free paper towels
  Plastic sheets

## 4.0 Water-Level Measurement

### 4.1 Introduction

Prior to obtaining a water-level measurement, cut a slit in one side of the plastic sheet and slip it over and around the well, creating a clean surface onto which the sampling equipment can be positioned. This clean working area should be a minimum of eight feet square. Care will be taken not to kick, transfer, drop, or in any way let soil or other materials fall onto this sheet unless it comes from inside the well. Do not place meters, tools, equipment, etc. on the sheet unless they have been cleaned first with a clean rag.

After unlocking and/or opening a monitoring well, the first task will be to obtain a water-level measurement. Water-level measurements will be made using an electronic or mechanical device. Electronic measurement devices will be used in all wells wherein a clearly audible sound cannot be produced with a mechanical device.

4.2 Well Security

Unlock and/or open the monitoring well. Enter a description of condition of the security system and protective casing on the Ground-Water Sample Collection Record shown in Table 1.

4.3 Measuring Point

Establish the measuring point for the well. The measuring point location should be clearly marked on the outermost casing or identified in previous sample collection records. If no measuring point can be determined, a measuring point should be established. The measuring point should be a point which is, or can easily be transposed vertically to, the survey control point for the well. Typically the top of the protective or outermost well casing will be used as the measuring point. The measuring point location should be described on the Ground-Water Sample Collection Record.

4.4. Measurement

To obtain a water-level measurement lower a decontaminated mechanical or an electronic sounding unit into the monitoring well. Care must be taken to assure that the water-level measurement device hangs freely in the monitoring well and is not adhering to the wall of the well casing. The water-level measuring tape will be lowered into the well until the audible sound of the unit is detected or the light on an electronic sounder illuminates. At this time the precise measurement should be determined by repeatedly raising and lowering the tape to converge on the exact measurement. The water-level measurement should be entered on the Ground-Water Sample Collection Record.

4.5 Decontamination

The measurement device shall be decontaminated immediately after use. Two persons are usually required to perform decontamination. Two handfuls of chemical-free paper towels will be obtained and one of which will be soaked with methanol. As the tape or line is rolled back onto the reel by one person, the second will wipe all free liquids and moisture from the tape or line with one towel closest to the reel, and follow with a second wipe of methanol a few inches behind.

5.0 Purge-Volume Computation

All monitoring wells to be purged prior to sample collection. Depending upon the ease of purging, 4 to 10 volumes of ground water present in a well shall be withdrawn prior to sample collection. The

volume of water present in each well shall be computed using the two measurable values, length of water column and monitoring well inside diameter. The water column length shall be computed as shown in Item 2b of Table 1. The monitoring-well diameter may be obtained by direct measurement in the field or from the boring log. Figures 1(a) and 1(b) will be used to compute the well volume. The one (1) well volume shall be multiplied by the appropriate factor (i.e. 4-10) to obtain the total purge volume.

## 6.0 Applications of Well-Purging and Sample-Collection Methods

### 6.1 Introduction

Purging must be performed for all ground-water monitoring wells prior to sample collection. The following sections explain the procedures to be used to purge and collect samples from monitoring wells.

Three general methods are used for well purging. Well purging may be achieved using bailers, surface pumps, or down-well submersible pumps.

In all cases pH and/or specific conductance will be monitored during purging. Field parameter values will be entered on the Ground-Water Sample Collection Record along with the corresponding purge volume.

### 6.2 Bailing

In many cases bailing may be the most convenient method for well purging. The cost of bailers and their relative size allows that many be transported easily to be available for a field sampling program so that it is not necessary to decontaminate or clean bailers between sample points. The small size of bailers allows that complete cleaning be performed without extensive decontamination facilities. ERT typically uses thin wall Teflon® bailers which are translucent, and can be readily disassembled for cleaning. These bailers contain approximately one liter (3.78 liters = 1 gallon) when full.

Bailing presents two potential problems with well purging. First, increased suspended solids may be present in samples as a result of the turbulence caused by raising and lowering the bailer through the water column. High solids concentrations may require that total suspended solids (TDS) and the chemical character of solids be evaluated during sample analyses. Second, bailing may not be feasible for wells which require that greater than twenty (20) gallons be removed during purging. Such bailing conditions

mandate that long periods be spent during purging and sample collection. All ground-water collected from monitoring wells for subsequent volatile organic analyses shall be collected using bailers, regardless of the purge method.

## 6.3 Surface Pumping

Ground-water withdrawal using pumps located at the ground surface is commonly performed with centrifugal or peristaltic pumps.

All applications of surface pumping will be governed by the depth to the ground-water surface. Peristaltic and centrifugal pumps are limited to conditions where ground water need only be raised through approximately 20-25 feet of vertical distance. The lift potential of a surface pumping system will depend upon the net positive suction head of the pump and the friction losses associated with the particular suction line.

Surface pumping can be used for many applications of well purging and ground-water sample collection. In all cases, pumping cannot be used for the collection of samples to be analyzed for volatile organic compounds.

### 6.3.1 Peristaltic Pump

Peristaltic pumps provide a low rate of flow typically in the range of 0.02-0.2 gallons/min (75-750 ml/min). For this reason, peristaltic pumps are not particularly effective for well purging. Peristaltic pumps are suitable for purging situations where a relatively long time is available for purging. Peristaltic pumps will lift water a maximum of approximately 20 - 25 feet. Peristaltic pumps are most often used for the field filtering of samples and therefore they are most often used to obtain water samples, from purged monitoring wells for direct filtration.

### 6.3.2 Centrifugal Pump

Centrifugal pumps are designed to provide a high rate of pumping, in the range of 10-40 gallons per minute (gpm). Centrifugal pumps can be used to pump at lower rates (1-5 gpm) if friction losses in the suction line are large, the pump drive motor is maintained at low speeds, or a valve is used to regulate discharge.

When centrifugal pumps are used, samples will be obtained from the suction line during pumping by an entrapment scheme as shown in Figure 2. While pumping is ongoing the

containment valves will be closed and immediately thereafter the pump will be shut-off. The breather plug will then be opened and the sample obtained from the stopcock. This method will not be used for the collection of samples for analyses of volatile organic compounds.

Two methods, direct connection or down well suction line may be used for well purging and/or sample collection by centrifugal pumps.

Table 2 provides a summary of the advantages, disadvantages and applications for each of the two methods.

6.3.2.1 Direct Connection

The Direct Connection method is used to collect ground-water samples with centrifugal pumps. As with all pumping methods sample turbulence precludes the use of pumping for the collection of samples for analyses of volatile organic compounds.

Direct Connection requires that a suction line system be constructed which will allow that sample collection be performed on the suction side of the pump so that sample contamination due to pump contact is eliminated. In addition, the valve system on the suction line will provide a mechanism for the control of pumping. Each time pumping stops a value will be closed immediately to prevent the return of water to the well which has contacted the pump.

6.3.2.2 Down-Well Suction Line

Down-well suction lines are used where direct connection cannot be made to the well riser pipe. Down-well suction lines are used for applications when purging should include raising and lowering the suction tubing throughout the entire length of the water column.

The down-well suction line method basically requires that a continuous length of tubing be used from the pump to the end of suction line. For this reason, the method is only used for well purging because samples can only be collected from the discharge side of the pump.

6.3.3 Submersible Pump

Submersible pumps provide an effective means for well purging and in some cases sample collection. Submersible pumps are particularly useful for situations where the depth to water table is greater than twenty (20-30) feet and the depth or diameter of the well requires that a large purge volume be removed during purging. Submersible pumps also provide a continuous discharge which allows that less variability be encountered with samples collected by this method.

ERT uses the Johnson-Keck pump model SP-81 which has a 1.75 inch diameter pump unit. The pump diameter restricts use to monitoring wells which have inside diameters equal to or greater than two (2) inches. As with other pump-type purge/sample collection methods, submersible pumps will not be used for the collection of samples for analyses of volatile organic compounds.

7.0 Purging- and Sample-Collection Procedures

7.1 Bailing

7.1.1 Obtain a clean/decontaminated bailer and a spool of polypropylene rope or equivalent bailer cord. Using the rope at the end of the spool tie a bowline knot or equivalent through the bailer loop. Test the knot for adequacy by creating tension between the line and the bailer. Tie again if needed.

7.1.2 Remove the aluminum foil wrapping from the bailer, and, while holding the bailer, place it inside the well to verify that an adequate annulus is present between the bailer and the well casing to allow free movement of the bailer.

7.1.3 Lower the bailer to the bottom of the monitoring well and remove an additional five feet of cord from the spool. Cut the cord at the spool and secure the rope to the well head or the wrist of the person who shall perform the bailing.

7.1.4 Raise the bailer by grasping a section of cord using each hand alternately. This bailer lift method will provide that all of the bailer cord will not come into contact with the ground or other potentially contaminated surfaces.

7.1.5 Bailed ground water will be poured from bailer into a graduated bucket to measure the purged water volume.

7.1.6 During sample collection bailers will be lowered to the bottom of monitoring wells and withdrawn slowly through the entire water column.

7.1.7 Samples collected by bailing will be poured directly into sample containers from bailers which are full of fresh ground water. During sample collection, bailers will not be allowed to contact the sample containers.

7.2 Peristaltic Pump

7.2.1 Place a new suction and discharge line in the peristaltic pump. Silicon tubing must be used through the pump head. A second type of tubing may be attached to the silicon tubing to create the suction and discharge lines. Such connection is advantageous for the purpose of reducing tubing costs, but only be done if airtight connections can be made. Tygon tubing will not be used when performing well purging or collecting samples for organic analysis. The suction line must be long enough to extend to the static ground-water surface and reach further should drawdown occur during pumping.

7.2.2 Measure the length of the suction line and lower it down the monitoring well until the end is in the upper 2-5 inches of the water column present in the well. Start the pump and direct the discharge into a graduated bucket.

7.2.3 Measure the pumping rate in gallons per minute by recording the time required to fill a selected volume of a bucket. Flow measurement shall be performed three times to obtain an average rate.

7.2.4 The pumping shall be monitored to assure continuous discharge. If drawdown causes the discharge to stop, the suction line will be lowered very slowly further down into the well until pumping restarts. The suction line will be lowered to assure that the end of the suction line is maintained in the uppermost 2-5 inches of the water column.

7.2.5 Measurments of pH and specific conductance will be made periodically during well purging. All readings will be entered on the Ground-Water Sample Collection Record.

7.2.6 Samples will be collected after the required purge volume has been withdrawn and the field parameters (pH and Specific Conductance) have stabilized.

7.2.7 When the sample bottles are prepared, each shall be filled directly from the discharge line of the peristaltic pump. Care will be taken to keep the pump discharge line from contacting the sample bottles. Ground-water samples requiring filtration prior to placement in sample containers, will be placed in intermediate containers for subsequent filtration or filtered directly using the peristatic pump.

7.2.8 At each monitoring point when use of the peristaltic pump is complete, all tubing including the suction line, pump head and discharge line must be disposed of. In some cases where sampling will be performed frequently at the same point, the peristaltic pump tubing may be retained between each use in a clean zip-lock plastic bag.

7.3 Centrifugal Pump

7.3.1 Direct Connection Method

7.3.1.1 Establish direct connection to the monitoring well using pipe connections, extensions, and elbows, with Teflon® tape wrapping on all threaded connections. If the centrifugal pump will subsequently be used for sample collection, a sample isolation chamber will be placed in the suction line configuration as shown in Figure 2.

7.3.1.2 Prime the pump by adding tap water to the pump housing until the housing begins to overflow.

7.3.1.3 Start the pump and direct the discharge into a graduated bucket or a bucket of known capacity (>2.5 gallons).

7.3.1.4 Measure the pumping rate in gallons per minute by recording the time required to fill a selected volume of a bucket. Flow measurement should be performed three times to obtain an average rate. Pumping will be observed at all times to determine if pumping rates are continuous, fluctuating, or diminishing. If discharge stops, the pump will be throttled back to determine if pumping will restart at a lower rate. If pumping does not

restart, the pump should be shut off to allow the well to recharge.

7.3.1.5 Measurements of pH and specific conductance will be made periodically during well purging. All readings will be entered on the Ground-Water Sample Collection Record.

7.3.1.6 Samples will be collected after the required purge volume has been withdrawn and the field parameters (pH and Specific Conductance) have stabilized.

7.3.1.7 While pumping is on-going the containment valves in the Sample Isolation Chamber will be closed and the pump immediately shut off.

7.3.1.8 When the sample bottle is prepared, the breather plug will be removed and the stopcock at the sample collection point opened and the sample bottle filled.

7.3.1.9 At each monitoring well when use of a centrifugal pump is complete, all suction line parts will be decontaminated in accordance with the SOP for Decontamination.

7.3.2 Down-Well Suction-Line Method

7.3.2.1 Lower a new suction line into the well. The suction line will have a total length at least great enough to extend to the water table and account for a minimum of five (5) feet of drawdown. Note should be made that drawdown may exceed the depth where pumping will terminate as a result of a limitation derived from suction-line conditions and the lift potential of the pump. All connections will be made using Teflon® ferrules and Teflon® thread wrapping tape.

7.3.2.2 Prime the pump by adding tap water to the pump housing until the housing begins to overflow.

7.3.2.3 Start the pump and direct the discharge into a graduated bucket or a bucket of known capacity (>2.5 gallons).

7.3.2.4 Measure the pumping rate in gallons per minute by recording the time required to fill a selected

volume of a bucket. Flow measurement should be performed three times to obtain an average rate. Pumping will be observed at all times to determine if pumping rates are continuous, fluctuating or diminishing. If discharge stops, the pump will be throttled back to determine if pumping will restart at a lower rate. If pumping does not restart, the pump should be shut off to allow the well to recharge.

The rate of recharge will be measured by inserting water-level measurement equipment down the well to determine approximately at what rate the well is recharging, and therefore when pumping may be restarted. All information pertaining to discharge conditions and recharge rates will be entered on the Ground-Water Sample Collection Record.

7.3.2.5 Measurements of pH and specific conductance will be made periodically during well purging. All readings will be entered on the Ground-Water Sample Collection Record.

7.3.2.6 The valve at the pump on the line will be closed whenever pumping terminates or pumping is stopped. This practice will minimize the return to the well of water which has contacted the inside of the pump housing.

7.3.2.8 At each monitoring well when use of a centrifugal pump is complete all suction line tubing will be disposed of.

7.4 Submersible Pump

7.4.1 Prior to using a submersible pump, a check will be made of well diameter and alignment. A 1.75 inch diameter decontaminated cylindrical tube will be lowered to the bottom of each monitoring well to determine if the alignment or plumbness of a well is adequate to accommodate the submersible pump. The well alignment survey may also be used to determine the total depths of wells. All observations will be entered in the Ground-Water Sample Collection Record.

7.4.2 Slowly lower the submersible pump into the monitoring well taking notice of any roughness or restrictions within the riser.

7.4.3 Count the graduations on the pump discharge line and stop lowering when the stainless steel portion is below the uppermost section of the static water column within monitoring well. Secure the discharge line and power cord to the well casing.

7.4.4 Connect the power cord to the power source (i.e., rechargeable battery pack or auto battery monitor) and turn the pump on (forward mode). When running, the pump can usually be heard by listening near the well head.

7.4.5 Voltage and amperage meter readings on the pump discharge will be checked continuously. The voltage reading will decline slowly during the course of a field day representing the use of power from the battery. Amperage readings will vary depending upon the depth to water table. Amperage readings greater than 10 amps usually indicate a high solids content in the ground water in which cases pump clogging will most likely occur. If a steady increase in amperage is observed, the pump should be shut off, allowed to stop, switched to the reverse mode, stopped again and then placed in forward mode. If high amperage readings persist, the pump should be withdrawn and checked using the large upright cylinder and tap water. Ground-water conditions such as high solids may require that an alternate purge/sample method be used.

7.4.6 Drawdown will be monitored continuously by remaining near the well at all times and listening to the pump. When drawdown occurs, a metallic rotary sound will be heard as the pump intake becomes exposed and ceases to discharge water, but continues to run. The pump will be lowered immediately to continue pumping water within the uppermost section of the static water column. NOTE: The submersible pump will not be allowed to run while not pumping for more than five seconds.

If drawdown continues to the extent that the well is pumped dry, the well will be allowed to recharge. The rate of recharge will be approximately determined by re-starting the submersible pump after a ten (10) minute period with the pump off. The pumping rate shall be re-measured and/or the total discharge volume collected to determine the recharge volume.

7.4.7 Direct the pump discharge to a graduated bucket or a bucket of known capacity.

7.4.8 Measure the pumping rate in gallons per minute by recording the time required to fill a selected volume of a bucket. Flow measurement shall be performed three times to obtain an average rate. The performance of a submersible pump will be observed at all times by at least one field worker.

7.4.9 Measurements of pH and specific conductance will be made periodically during well purging. All readings and respective purge volumes will be entered on the Ground-Water Sample Collection Record.

7.4.10 Samples will be collected after the required purge volume has been withdrawn and the field parameters (pH and specific conductance) have stabilized.

7.4.11 While pumping is on-going and when sample bottles are prepared, bottles will be filled directly from the discharge line of the pump taking care not to touch sample bottles to the discharge line.

7.4.12 At each monitoring well when use of the submersible pump is complete, the pump, discharge line and power cord shall be decontaminated according to the procedures contained in the SOP for Decontamination.

## 8.0 Sample Preparation

### 8.1 Introduction

Prior to transport or shipment, ground-water samples may require preparation and/or preservation. Field preparation may entail filtration, or preservation in the form of chemical additives or temperature control.

Specific preservation techniques are described in the EPA document, Handbook for Sampling and Sample Preservation of Water and Wastewater (EPA-600/4-82-029). The EPA manual will be consulted during the planning stage of the project. Project-specific sampling plans shall be assembled using the approved procedures obtained from the EPA manual.

### 8.2 Filtration

Ground-water samples collected for dissolved metals analyses will be filtered prior to being placed in sample containers. Ground-water filtration will be performed using a peristaltic pump and a 0.45 micron, water filter. Typically the water filters are 142 mm in diameter and are usually placed in 142 mm polycarbonate housings.

The filtration of ground-water samples shall be performed either directly from the monitoring well or from intermediate sample containers such as decontaminated buckets. In either case, well purging shall be performed first. Fresh ground water shall then be filtered and discharged from the filtration apparatus directly into sample containers. For most dissolved metal analyses, pH adjustment of the sample is required and shall be performed after filling the sample bottles.

## 9.0 Documentation

A number of different documents will be completed and maintained as a part of ground-water sampling. The documents will provide a summary of the sample-collection procedures and conditions, shipment method, the analyses requested and the custody history. The list of documents is:

- Ground-water sample collection record
- Sample labels
- Chain of custody
- Shipping receipts

Sample labels shall be completed at the time each sample is collected and will include the information listed below. A sample label is shown in Figure 3.

- Client or project name
- Sample number
- Designation (i.e., identification of sample point no.)
- Analysis
- Preservative (e.g., filtration, acidified pH<2 $HNO_3$)
- Sample-collection date
- Sampler's name

Figure 4 displays the chain of custody record used by ERT. The chain of custody form is the record sample collection and transfer of custody. Information such as the sample collection date, sample identification and origination, client or project name shall be entered on each chain of custody record. In accordance with 40 CFR 261.4(d) the following information must accompany all ground water samples which are known to be non-hazardous and to which U.S. Department of Transportation and U.S. Post Office regulations do not apply. Such information is:

- sample collector's name, mailing address and telephone number,
- analytical laboratory's name, mailing address and telephone number,
- quantity of each sample,
- date of shipment, and
- description of sample.

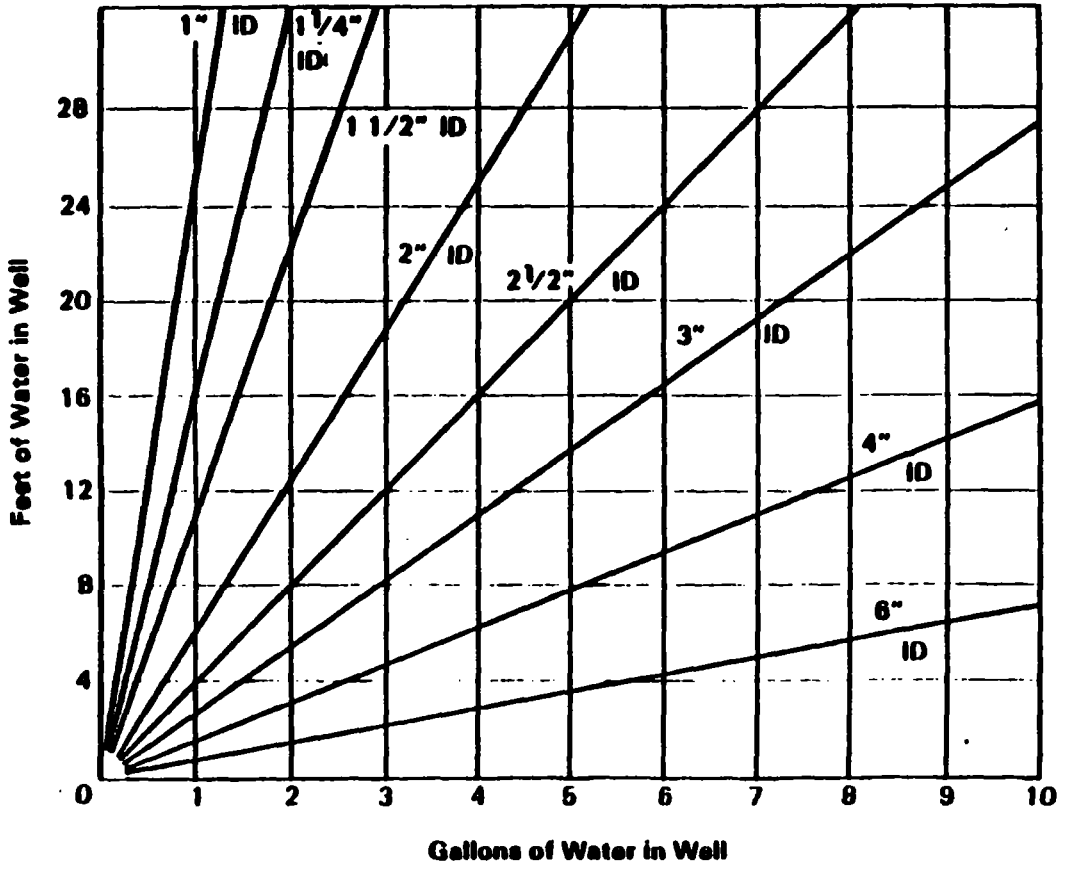
The chain of custody forms provide a location for entry of the above-listed information.

## 10.0 References

EPA, Handbook for Sampling and Sample Preservation of Water and Wastewater EPA-600/4-82-029, September 1982.

Geotrans, Inc. RCRA Permit Writer's Manual, Ground-Water Protection prepared for U.S. EPA. Contract No. 68-01-6464, October 1983.

Code of Federal Regulations, Chapter 40 (Section 261.4(d).




(a) Graphical Explanation

| Volume/Linear Ft. of Pipe | | |
|---|---|---|
| ID(in) | Gal | Liter |
| 1/4 | 0.003 | 0.010 |
| 3/8 | 0.006 | 0.022 |
| 1/2 | 0.010 | 0.039 |
| 3/4 | 0.023 | 0.087 |
| 1 | 0.041 | 0.154 |
| 2 | 0.163 | 0.618 |
| 3 | 0.367 | 1.39 |
| 4 | 0.653 | 2.47 |
| 6 | 1.47 | 5.56 |

(b) Volume Factors

Figure 1 Purge Volume Computation

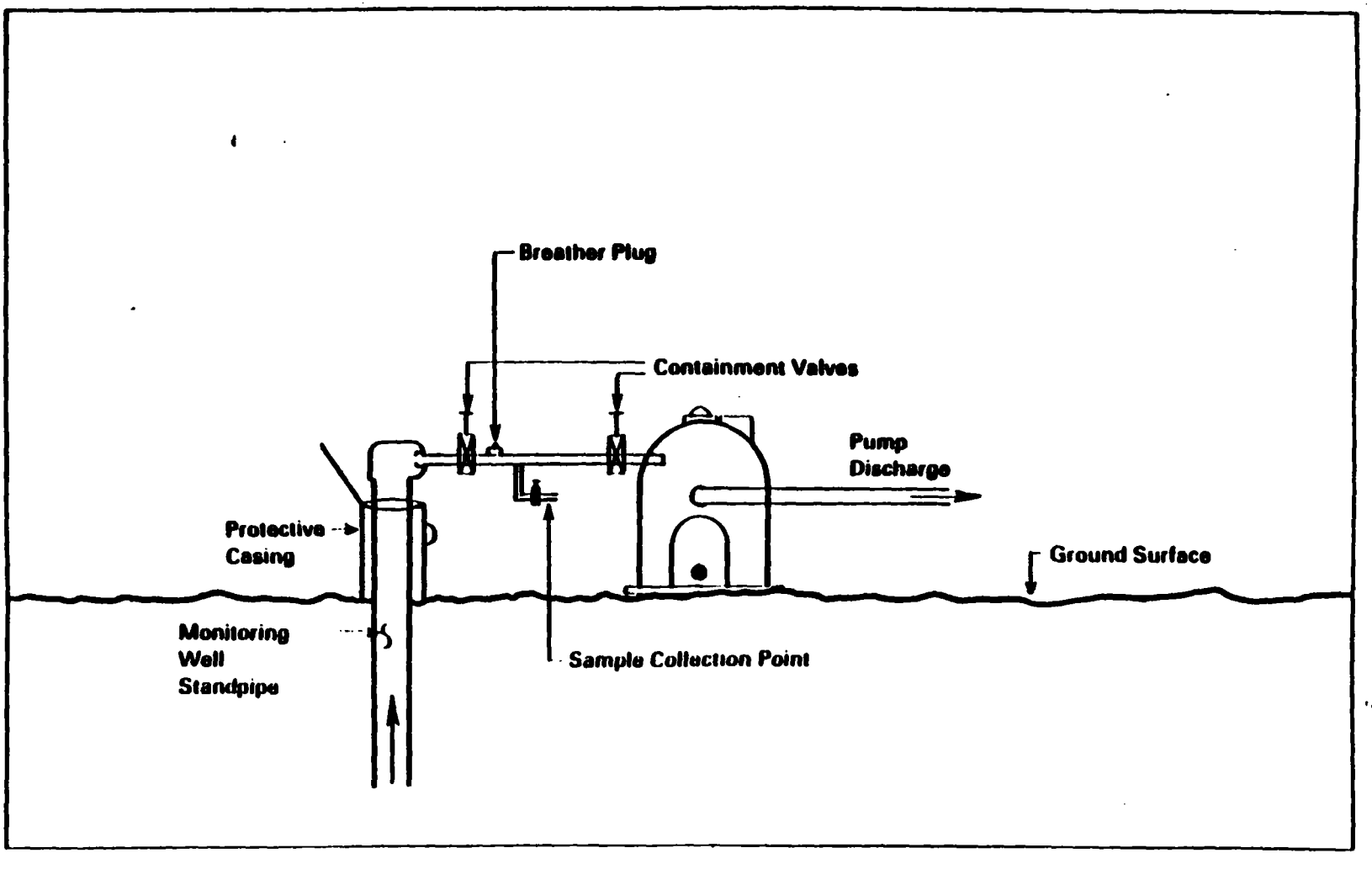



Figure 2 Down Well Suction Line Configuration

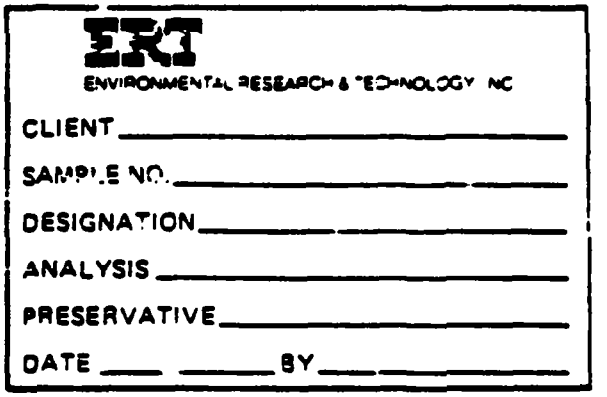



Figure 3 Sample Container Label

ERT STANDARD OPERATING PROCEDURE

Number: 7320 Date of Issue: March 12, 1984

Title: Calibration and Operation of Hydrolab Water Quality Monitor

| Organizational Acceptance | Authorization | Date |
|---|---|---|
| Originator | | |
| Department Manager | | 3/13/84 |
| Divisional Manager | | |
| Group Quality Assurance Officer | | |
| Other | | |

| Revisions | Changes | Authorization | Date |
|---|---|---|---|

# STANDARD OPERATING PROCEDURE

Title: Calibration and Operation of Hydrolab Water Quality Monitor

Date: 1st Qtr 1984
Number: SOP 7320
Revision: 1

## 1.0 Applicability

This Standard Operating Procedure (SOP) provides basic instructions to be employed for the field operation of Hydrolab digital multimeters (Model Nos. 4041 and 8000). Hydrolabs are used for field measurement of water-quality parameters.

## 2.0 Responsibilities

The field team is responsible for ensuring that the Hydrolab unit is in proper operating condition prior to use in the field. All system-calibration checks are the responsibility of the field team.

## 3.0 Materials

- Hydrolab Operation and Maintenance Instruction Manual
- Hydrolab Sonde unit, battery pack and surface unit
- Hydrolab calibration-cup
- Two Fisher-brand laboratory potassium chloride (KCl) standard solutions (known conductivity at 25°C)
- Two freshly prepared pH buffer solutions. Generally pH 7.0 and pH 4.0 or 10.0 are used.
- Distilled or de-ionized water (approximately two liters)
- Chemical-free paper towels
- Screwdrivers (as supplied in the Hydrolab Accessory Kit)

## 4.0 Procedures

The Hydrolab provides simultaneous measurement of four water quality parameters; 1) dissolved oxygen, in mg/l, 2) temperature, in °C; 3) pH, in standard units, and 4) conductivity, in umhos/cm (uS/cm). The panel switch on the front of the indicator unit controls which parameter is being measured and read-out.

The display is read in the following manner; temperature, pH and dissolved oxygen are read out directly. For example, a temperature of 21.8°C will be displayed as 21.8. A dissolved oxygen (D.O.) or pH reading of 8.1 will be displayed at 08.1. Conductivity is read out directly on the 2k scale. If the 20k scale is required to measure higher conductivity the number that is displayed will need to be

Are all these to be measured in the field? It is not specified.

multiplied by 10. In the 200k scale the reading will be multiplied by 100. For example, suppose the sample being measured has a conductivity of 1527 uS/cm. Using the 2k scale, the display will show 1527 (direct read-out). Using the 20k scale the display will show 153 (153 x 10 = 1530 uS/cm). Using the 200k range the display will show 015 (015 x 100 = 1500 uS/cm). Only the Hydrolab model 4041 offers the three scale measurement. The Hydrolab model 8000 is restricted to measurement of conductivity within the range of 0-2000.

4.1 Hydrolab Calibration

A complete calibration check should be performed before going to and after returning from a field sampling/water quality measurement activity. The calibration procedures should be carried out in a controlled environment such as a laboratory, but a field office or closed-in shelter may also be used.

At least one hour prior to calibration, take the following preparatory steps:

1) Remove the "Storage-Cup" from the Sonde Unit.

2) Remove the protective guard from the dissolved oxygen sensor.

3) Install the "Calibration-Cup" on the Sonde Unit and fill to the brim with distilled water.

4) Seal the Calibration Cup with the soft plastic cap and store the sonde unit, calibration standards, and the distilled water at constant room temperature for at least one hour in order to bring the various sensors, temperature compensating elements, and the calibration solutions into thermal equlibrium (within a few degrees).

All of the calibration controls are located on the front panel of the Indicator Unit. Adjustments, if necessary, should be made in the following manner:

1) Remove the appropriate seal-screw for the parameter being adjusted.

2) Insert a small screwdriver through the access hole and adjust the calibration control in the direction which brings the reading into agreement with the value of the standard solution being employed.

3) Replace the seal-screw.

A RINSE STEP will be used several times during the calibration procedure. It is to be performed in the following manner: Fill the calibration cup halfway with de-ionized or distilled water. Snap on the soft plastic cap; shake the sonde unit for ten seconds and then pour out the water. Repeat twice more using fresh de-ionized or distilled water. Remove the cup and shake as much of the rinse water as possible from the electrodes.

4.1.1 Dissolved Oxygen Calibration

The Dissolved Oxygen system is the first to be calibrated since the water that has been stored in the calibration cup is used to maintain control of the temperature inside the cup. The calibration standard is either a water sample of a known D.O. concentration (determined in the laboratory by the Winkler or iodemtric method in accordance with Standard Methods for the Examination of Water and Wastewater, 15th Edition, APHA-AWWA-WPCF, 1980 or water-saturated air at the temperature inside the calibration cup. The following procedures are for the water-saturated air method for D.O. calibration.

Invert the Sonde Unit and remove the soft plastic cap. Pour off enough water to bring the level to just below the D.O. membrane- retainer O-ring. With a clean paper towel or tissue blot any moisture from the D.O membrane. Cover the calibration cup mouth with one of the hard plastic caps provided in the Accessory Kit. This will keep drafts from blowing on the membrane. Do not seal the cup with the plastic cap, because that could cause a partial-pressure change in the cup. Wait approximately 5 minutes, or until the reading is stable, then switch to the TEMPERATURE position and record the temperature reading. Refer to Table 1 for the correct oxygen concentration at this temperature. Since the table values refer to concentrations at Standard Pressure it will be necessary to correct the value for local barometric pressure. This should be done in the following manner:

Correct D.O. Setting = (Local Barometric Pressure/760mm) x (Table value at Cup Temperature)

EXAMPLE: If T = 28.5°C and Local Barometric Pressure = 800mm,

Correct D.O. Setting = (800mm/760mm) x (7.6 mg/l)
= 8.0 mg/l

If a barometer is not available, the equivalent pressure may be estimated from Table 2 which relates atmospheric pressure with elevation above mean sea level. Therefore, the approximate atmospheric pressure at an altitude of 2000 feet, for example, would be: Local Atmospheric Pressure = 705mm Hg.

Adjust the Dissolved Oxygen calibration control until the proper value (rounded to nearest tenth) is displayed. Pour our the water; and then follow with a RINSE STEP.

4.1.2 pH Calibration

Calibrating the pH system requires the use of two Fisher-brand pH laboratory buffer solutions. Depending upon the application, either pH 4.0 or pH 10.0 is used in addition to pH 7.0. Invert the sonde unit and fill the calibration cup with fresh pH 7.0 buffer solution. Switch to "pH", and wait approximately 5 minutes for thermal equilibrium. Then adjust the pH calibration control until 7.0 is displayed on the read-out.

Pour out the 7.0 buffer and repeat the RINSE STEP. Invert the sonde unit and screw on the calibration cup; fill with 10.0 or 4.0 buffer. After approximately 5 minutes, adjust the pH "Slope" control until either 10.0 or 4.0 (as appropriate for the buffer being used) is displayed on the read-out. Pour out the buffer and repeat the RINSE STEP Two Times

4.1.3 Conductivity Calibration

After the second RINSE STEP, take a clean paper towel or tissue, and blot most of the moisture in the electrode area so that the standard will not suffer dilution.

Install a clean calibration cup and invert the sonde unit. The conductivity system is calibrated using at least two prepared KCl standard solutions with a known conductivity at 25°C. From Table 3, select two standard solutions with values of approximately one-third and two-thirds of the range you are most likely to encounter in the field. For example, if you are going to be working in fresh water (0-2K scale) you would want to use a 0.01M standard and a 0.005M standard. Select the more concentrated of the two standards and pour it slowly down the side of the calibration cup until full. When the reading is stable, adjust the conductivity calibration control until the display matches

the value listed in Table 3. Empty the calibration cup and repeat the RINSE STEP Two Times. Pour in the second standard. Check the reading on the Display. It should be correct within ± 1% of the range being used. For example, if the 0-2K scale is used, the reading for the second standard should be correct within ± 20 uS/cm of the true value. Pour out the standard solution. Perform a RINSE STEP.

4.1.4 Temperature Calibration

The temperature system is factory calibrated and is accurate to ± 0.2°C. No calibration adjustment is provided. A periodic check of the temperature system against an NBS-traceable thermometer should be performed as a verification.

4.2 Final Preparation

Turn the system off and disconnect the system components. Replace all rubber dust caps. Remove the Calibration Cup from the Sonde Unit and replace the protective guard on the dissolved oxygen electrode. Fill the Storage Cup with tap water and install onto the Sonde Unit. The system is now calibrated and ready for field use.

4.3 Field Operation

Remove the Storage Cup from the calibrated sonde unit and install the guard or the optional sample circulator. Connect the system components. Lower the sonde unit into the water (sideways, if possible) and shake it to dislodge air bubbles trapped in the conductivity cell block. Release the sonde unit and lower it to sample depth. Wait until the readings stabilize (D.O. is the best indicator) and then record the value for each parameter. Repeat at new depths or locations.

When using for ground water sampling, pour/place a sample of ground water into the Storage Cup and attach it to the sonde so that all nodes are submerged.

Check the battery voltage occasionally; charge or change batteries if the level drops below 10.5 volts. DO NOT charge the battery routinely after each day's use. Doing so may shorten the life of the battery. Use the battery until the voltage level drops to between 10.5 and 11.0 volts. At this point put the battery on charge for 24 hours.

TABLE 1

DISSOLVED OXYGEN SATURATION VALUES IN DISTILLED WATER AT 760 mm Hg

| Temp. (°C) | DO (mg/l) | Temp (°C) | DO (mg/l) |
|---|---|---|---|
| 0.0 | 14.6 | 15.5 | 9.9 |
| 0.5 | 14.4 | 16.0 | 9.8 |
| 1.0 | 14.2 | 16.5 | 9.7 |
| 1.5 | 14.0 | 17.0 | 9.6 |
| 2.0 | 13.9 | 17.5 | 9.5 |
| 2.5 | 13.7 | 18.0 | 9.4 |
| 3.0 | 13.5 | 18.5 | 9.3 |
| 3.5 | 13.3 | 19.0 | 9.2 |
| 4.0 | 13.1 | 19.5 | 9.1 |
| 4.5 | 13.0 | 20.0 | 9.0 |
| 5.0 | 12.8 | 20.5 | 8.9 |
| 5.5 | 12.6 | 21.0 | 8.8 |
| 6.0 | 12.5 | 21.5 | 8.8 |
| 6.5 | 12.3 | 22.0 | 8.7 |
| 7.0 | 12.1 | 22.5 | 8.6 |
| 7.5 | 12.0 | 23.0 | 8.5 |
| 8.0 | 11.8 | 23.5 | 8.4 |
| 8.5 | 11.7 | 24.0 | 8.3 |
| 9.0 | 11.6 | 24.5 | 8.2 |
| 9.5 | 11.4 | 25.0 | 8.2 |
| 10.0 | 11.3 | 25.5 | 8.1 |
| 10.5 | 11.1 | 26.0 | 8.0 |
| 11.0 | 11.0 | 26.5 | 8.0 |
| 11.5 | 10.9 | 27.0 | 7.9 |
| 12.0 | 10.8 | 27.5 | 7.8 |
| 12.5 | 10.6 | 28.8 | 7.7 |
| 13.0 | 10.5 | 28.5 | 7.6 |
| 13.5 | 10.4 | 29.0 | 7.6 |
| 14.0 | 10.3 | 29.5 | 7.5 |
| 14.5 | 10.2 | 30.0 | 7.4 |
| 15.0 | 10.0 | 30.5 | 7.4 |

TABLE 2

| Site Elevation (Feet above mean sea level) | Approximate Mean Barometric Pressure (mm Hg) |
|---|---|
| 1000 | 733 |
| 1500 | 720 |
| 2000 | 705 |
| 2500 | 694 |
| 3000 | 680 |
| 3500 | 669 |
| 4000 | 656 |
| 4500 | 644 |
| 5000 | 632 |
| 5500 | 620 |
| 6000 | 609 |
| 6500 | 598 |
| 7000 | 586 |
| 7500 | 575 |
| 8000 | 564 |
| 8500 | 554 |
| 9000 | 543 |
| 9500 | 533 |
| 10000 | 523 |

TABLE 3

CONDUCTIVITY CALIBRATION STANDARDS

| Conducitivies of Potassium Chloride Solutions at 25°C M.W. = 74.555 | | | Conductivity Reading on Hydrolab Display for Given Range Setting (uS/cm) | | |
|---|---|---|---|---|---|
| Conc. | Grams KCl/L | uS/cm | (0-2K) | (0-20K) | (0-200K) |
| 0.0005 | 0.03728 | 73.9 | | - | - |
| 0.001 | 0.07456 | 147.0 | 147 | - | - |
| 0.002 | 0.1491 | 292.0 | 292 | - | - |
| 0.005 | 0.3728 | 717.8 | 718 | - | - |
| 0.01 | 0.7456 | 1.413K | 1413 | 141 | - |
| 0.02 | 1.491 | 2.767K | --- | 277 | - |
| 0.05 | 3.728 | 6.668K | --- | 667 | - |
| 0.1 | 7.456 | 12.90K | --- | 1290 | 129 |
| 0.2 | 14.911 | 24.82K | --- | --- | 248 |
| 0.5 | 37.278 | 58.64K | --- | --- | 586 |
| 1.0 | 74.555 | 111.9K | --- | --- | 1119 |

NOTES:

(1) Two conductivity standards are recommended for each range setting (boxed-in values). Calibration adjustments will be made first with the higher concentration and then with the lower concentration.

(2) Single dashes indicate ranges which are not recommended for calibration checks.

(3) The Hydrolab model 8000 is restricted to conductivity readings between 0-2000 µS/cm (0-2k) scale), therefore conductivity readings and thus calibration solutions within the 0-20k and 0-200k ranges will not apply.

# ERT STANDARD OPERATING PROCEDURE

Number: 7510 Date of Issue: March 12, 1984

Title: Packaging and Shipment of Samples

| Organizational Acceptance | Authorization | Date |
|---|---|---|
| Originator | [signature] | [illegible]84 |
| Department Manager | [signature] | 3/13/84 |
| Divisional Manager | | |
| Group Quality Assurance Officer | | |
| Other | | |

| Revisions | Changes | Authorization | Date |
|---|---|---|---|

STANDARD OPERATING PROCEDURE

Title: Packaging and Shipment of Samples

Date: 1st Qtr. 1984
Number: SOP 7510
Revision:

## 1.0 Applicability

This Standard Operating Procedure (SOP) is concerned with the presentation of protocols associated with the packaging and shipment of samples. Two general categories of samples exist: environmental samples consisting of air, water and soil; and waste samples which include non-hazardous solid wastes and hazardous wastes as defined by 40 CFR Part 261.

## 2.0 Responsibilities

It is the responsibility of the project manager to assure that the proper packaging and shipping techniques are entered into each project specific sampling plan. The site operations manager shall be responsible for the enactment and completion of the packaging and shipping requirements outlined in project specific sampling plans. The site operations manager shall be responsible to research, identify and follow all applicable U.S. Department of Transportation (DOT) regulations.

## 3.0 General Method

The objective of sample packaging and shipping protocol is to identify standard procedures which will minimize the potential for sample spillage or leakage and maintain field sampling program compliance with U.S. EPA and U.S. DOT regulations.

The extent and nature of sample containerization will be governed by the type of sample, and the most reasonable projection of the sample's hazardous nature and constituents. The EPA regulations (40 CFR Section 261.4(d)) specify that samples of solid waste, water, soil or air, collected for the sole purpose of testing, are exempt from regulation under the Resource Conservation and Recovery Act (RCRA) when all of the following conditions are applicable:

A. Samples are being transported to a laboratory for analysis;
B. Samples are being transported to the collector from the laboratory after analysis;
C. Samples are being stored (1) by the collector prior to shipment for analyses, (2) by the analytical laboratory prior to analyses, (3) by the analytical laboratory after testing but prior to return of sample to the collector or pending the conclusion of a court case.

Qualification for categories A and B above require that sample collectors comply with U.S. DOT and U.S. Postal Service (USPS) regulations or comply with the following items if U.S. DOT and USPS regulations are found not to apply:

STANDARD OPERATING PROCEDURE

Title: Packaging and Shipment of Samples

Date: 1st Qtr. 1984
Number: SOP 7510
Revision:

The following information must accompany all samples and will be entered on a sample specific basis on chain of custody records:

- sample collector's name, mailing address and telephone number,
- analytical laboratory's name, mailing address and telephone number,
- quantity of sample,
- date of shipment
- description of sample

In addition, all samples must be packaged so that they do not leak, spill or vaporize.

4.0 Method

4.1 Place plastic bubble wrap matting over the base and bottom corners of each cooler or shipping container as needed to manifest each sample.

4.2 Obtain a chain of custody record as shown in Figure 1 and enter all the appropriate information as discussed in Section 3.0 of this SOP. Chain of custody records will include complete information for each sample. One or more chain of custody records shall be completed for each cooler or shipping container as needed to manifest each sample.

4.3 Wrap each sample bottle individually and place standing upright on the base of the appropriate cooler, taking care to leave room for some packing material and ice or equivalent. Rubber bands should be used to secure wrapping, completely around each sample bottle.

4.4 Place additional bubble wrap and/or styrofoam pellet packing material throughout the voids between sample containers within each cooler.

4.5 Place ice or cold packs in heavy duty zip-loc type plastic bags, close the bags, and distribute such packages over the top of the samples.

4.6 Add additional bubble wrap/styrofoam pellets to fill the balance of the cooler or container.

4.7 Obtain two pieces of chain of custody tape as shown in Figure 2 and enter the custody tape numbers in the appropriate place on the chain of custody form. Sign and date the chain of custody tape.

4.8 To complete the chain of custody form enter the type of analysis required for each sample, by container, under the "ANALYSES" section. Under the specific analysis enter the quantity/volume of sample collected for each corresponding analysis.

If shipping the samples where travel by air or other public transportation is to be undertaken, sign the chain of custody record thereby relinquishing custody of the samples. Reliquishing custody should only be performed when directly transmitting custody to a receiving party or when transmitting to a shipper for subsequent receipt by the analytical laboratory. Shippers should not be asked to sign chain of custody records.

4.9 Remove the back carbon copy from the chain of custody record and retain with other field notes. Place the remaining copies in a zip-lock type plastic bag and place the bag on the top of the contents within the cooler or shipping container.

4.10 Close the top or lid of the cooler or shipping container and with another person rotate/shake the container to verify that the contents are packed so that they do not move. Improve the packaging if needed and reclose.

When travelling with samples by automobile, and where periodic changes of ice are required, the cooler should only be temporarily closed so that reopening is simple. In these cases, chain of custody will be maintained by the person transporting the sample and chain of custody tape will not be used.

4.11 Place the chain of custody tape at two different locations on the cooler or container lid and overlap with transparent packaging tape.

4.12 Packaging tape should be placed entirely around the sample shipment containers. A minimum of one to two full rotations of packaging tape will be placed at at least two places on the cooler. Shake the cooler again to verify that the sample containers are well packed.

4.13 If shipment is required, transport the cooler to an overnight express package terminal. Obtain copies of all shipment records as provided by the shipper.

4.14 If the samples are to travel as luggage, check with regular baggage.

STANDARD OPERATING PROCEDURE

Title: Packaging and Shipment of Samples

Date: 1st Qtr. 1984
Number: SOP 7510
Revision:

4.15 Upon receipt of the samples, the analytical laboratory will open the cooler or shipping container and sign as "received for laboratory" each chain of custody form. The laboratory will verify that the chain of custody tape has not been broken previously and that the chain of custody tape number corresponds with the number on the chain of custody record. The analytical laboratory will then forward the back copy of the chain of custody record to the sample collector to indicate that sample transmittal is complete.

## 5.0 Documentation

As discussed in Section 4.0 the documentation for supporting the sample packaging and shipping will consist of chain of custody records and shipper's records. In addition a description of sample packaging procedures will be written in the field log book. All documentation will be retained in the project files following project completion.

# CHAIN OF CUSTODY RECORD

| Client/Project Name | Project Location | ANALYSES |
|---|---|---|
| Project No. | Field Logbook No. | |
| Sampler: (*Signature*) | Chain of Custody Tape No. | |

| Sample No./ Identification | Date | Time | Lab Sample Number | Type of Sample | | | | | | | REMARKS |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | | | | | | | |
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| | | | | | |
|---|---|---|---|---|---|
| Relinquished by: (*Signature*) | Date | Time | Received by: (*Signature*) | Date | Time |
| Relinquished by: (*Signature*) | Date | Time | Received by: (*Signature*) | Date | Time |
| Relinquished by: (*Signature*) | Date | Time | Received for Laboratory: (*Signature*) | Date | Time |
| Sample Disposal Method: | Disposed of by: (*Signature*) | | | Date | Time |

**SAMPLE COLLECTOR**

Environmental Research and Technology, Inc.
696 Virginia Road
Concord, MA 01742
617-369-8910

**ANALYTICAL LABORATORY**

**ERT**

**No. 1661**

1974 3 84

Figure 1

ERT Date _____ Sig. _____ Nº 30432

Figure 2

ERT

# STANDARD OPERATING PROCEDURE

Number: 7600 Date of Issue: 1st Quarter, 1984

Title: Decontamination of Equipment

| Organizational Acceptance | Authorization | Date |
|---|---|---|
| Originator | [signature] | 3/3/84 |
| Department Manager | [signature] | 3/2/84 |
| Divisional Manager | [signature] | 3-2-84 |
| Group Quality Assurance Officer | [signature] | [illegible] |
| Other | | |

| Revisions | Changes | Authorization | Date |
|---|---|---|---|
| 1 | Update | [signature] | [illegible] |
| | | CE-M | 3-2-84 |
| | | AGC | 3/2/84 |
| | | EM | 3-2-84 |

## 1.0 General Applicability

This SOP describes the methods to be used for the decontaminization of all field equipment which becomes potentially contaminated during a sample collection task. The equipment may include split spoons, bailers, trowels, shovels, hand augers, or any other type of equipment used during field activities.

Decontamination is performed as a quality assurance measure and a safety precaution. It prevents cross-contamination between samples and also helps to maintain a clean working environment for the safety of all field personnel involved, including the environment.

Decontamination is mainly achieved by rinsing with liquids which include: soap and/or detergent solutions, tap water, deionized water, and methanol. Equipment will be allowed to air dry after being cleaned or may be wiped dry with chemical free cloths or paper towels if immediate re-use is needed.

The frequency of equipment use, dictates that most decontamination be accomplished at each sampling site between collection points. Waste products produced by the decontamination procedures such as waste liquids, solids, rags, gloves, etc. will be collected and disposed of properly based on the nature of contamination. All cleaning materials and wastes should be stored in a central location so as to maintain control over the quantity of materials used and/or produced throughout the study.

## 2.0 Responsibilities

It is the primary responsibility of the site operations manager to assure that the proper decontamination procedures are followed and that all waste materials produced by decontamination are properly stored and disposed of.

It is the responsibility of the project safety officer to draft and enforce safety measures which provide the best protection for all persons involved directly with sampling and/or decontamination.

It is the responsibility of any subcontractors (i.e., drilling contractors) to follow the proper, designated decontamination procedures that are stated in their contracts and outlined in the Project Health and Safety Plan.

It is the responsibility of all personnel involved with sample collection or decontamination to maintain a clean working environment and to ensure that any contaminants are not negligently introduced to the environment.

3.0 Supporting Materials

- cleaning liquids: soap and/or detergent solutions, tap water, deionized water, methanol
- personal safety gear (defined in Project Health and Safety Plan)
- chemical-free paper towels
- disposable gloves
- waste storage containers: drums, boxes, plastic bags
- cleaning containers: plastic buckets, galvanized steel pans
- cleaning brushes

4.0 Methods or Protocol for Decontamination

4.1 General Procedures

4.1.1 The extent of known contamination will determine to what extent the equipment needs to be decontaminated. If the extent of contamination cannot be readily determined, cleaning should be done according to the assumption that the equipment is highly contaminated until enough data are available to allow assessment of the actual level of contamination.

4.1.2 Adequate supplies of all materials must be kept on hand. This includes all rinsing liquids and other materials listed in Section 3.0.

4.1.3 The standard procedures listed in the following section can be considered the procedure for full field decontamination. If different or more elaborate procedures are required for a specific project, they will be spelled out in the project work plan. Such variations in decontamination may include following all, just part, or an expanded scope of the decontamination procedure stated herein.

4.2 Standard Procedures

4.2.1 Remove any solid particles from the equipment or material by brushing and then rinsing with available tap water. This initial step is performed to remove gross contamination.

4.2.2 Wash equipment sampler with the soap or detergent solution.

4.2.3 Rinse with tap water

4.2.4 Rinse with deionized water

4.2.5 Rinse with methanol

4.2.6 Repeat entire procedure or any parts of the procedure if necessary

4.2.7 Allow the equipment or material to air dry before re-using

4.2.8 Dispose of any soiled materials in the designated disposal container

## 5.0 Specific Decontamination Procedures

### 5.1 Submersible Pump

#### 5.1.1 Applicability

This procedure will be used to decontaminate submersible pumps between ground-water sample collection points and at the end of each day of use.

#### 5.1.2 Materials

- o plastic-nalgene upright cylinder
- o 5-10 gallon plastic water storage containers
- o methanol and dispenser bottle
- o deionized water and dispenser bottle
- o chemical free paper towels

5.1.3.1 During decontamination the submersible pump will be placed on a clean surface or held away from ground.

5.1.3.2 When removing the submersible pump from each well the power cord and discharge line will be wiped dry using chemical-free disposable towels.

5.1.3.3 Clean the upright plastic-nalgene cylinder with first a methanol and then a deionized water rinse, wiping the free liquids after each.

5.1.3.4 Reverse pump backwashing all removable residual water present in the pump tubing. The pump should be shut off as soon as intermittent flow is observed from the reverse discharge.

5.1.3.5 Rinse the stainless steel submersible down hole pump section with a liberal application of methanol and wipe dry.

5.1.3.6 Place the submersible pump section upright in the cylinder and fill the cylinder with tap water, adding 50-100 ml of methanol for every one liter of water.

5.1.3.7 Activate the pump in the forward mode withdrawing water from the cylinder.

5.1.3.8 Continue pumping until the water in the cylinder is pumped down and air is drawn through the pump. At this time air pockets will be observed in the discharge line. Shut off the pump immediately.

5.1.3.9 Remove the pump from the cylinder and place the pump in the reverse mode allowing that all removable water be discharged on to the ground surface as discussed in Step 2.

5.1.3.10 Using the water remaining in the cylinder, rinse the sealed portion of the power chord and discharge tube by pouring the water carefully over the coiled lines.

5.1.3.11 When reaching the next monitoring well place the pump in the well casing and wipe dry both the power and discharge lines with a clean paper towel as the pump is lowered.

5.1.4 Quality Assurance

To assure that decontamination is complete, field blank samples shall be collected using the cleaned submersible pump. These field blanks will be subsequently analyzed for the parameters of interest with respect to the ground water.

The procedure for collecting the field blanks will comprise using the pump to withdraw the tap water used for decontamination, from the plastic cylinder to sample containers. This field blank sample collection procedure shall only be performed after the materials to be used have been decontaminated.

# SECTION C

# HEALTH & SAFETY PLAN

SAFETY PLAN

for the

St. Louis Park Site
Well Monitoring Program
(Name of Site/Facility)

Located in

St. Louis Park (City) Minnesota (State)

Project Number: E415-100

Date: October 4, 1986

Prepared By: Kevin Powers

Date: 10/4/86

Approved By: _____
Project Manager

Date: _____

Kevin Powers
Health & Safety Manager

Date: 10/4/86

## SITE/PROJECT DESCRIPTION

SITE DESCRIPTION: ACTIVE? YES _____ NO X

An 80 acre site within the city of St. Louis Park, Minnesota was the former location of a coal tar distillation and wood treating facility. The facility is no longer active, however contribution of coal tar and creosote compounds to the City's groundwater is being investigated.

SCOPE OF PROJECT/TASK: To conduct an ongoing groundwater sampling program to determine the extent of groundwater contamination associated with the former coal tar distillation and wood treating plant.

PROPOSED ON-SITE ACTIVITIES: Sampling of various active pumping wells, inactive monitoring wells, piezometers and a granular activated carbon (GAC) facility stream within the St. Louis Park area.

PROPOSED DATE(S) OF FIELD ACTIVITY: To be determined

PERSONNEL REQUIREMENTS:

| NAME | RESPONSIBILITY |
|---|---|
| Bill Gregg | Coordinate/Conduct Sampling |
| Various St. Louis Park Personnel | Collect Samples |
| | |
| | |
| | |
| | |
| | |

# HAZARD EVALUATION

MATERIALS OF CONCERN: Coal tar and creosote compounds. Species present may include phenols and a wide array of polynuclear aromatic hydrocarbons such as Benzo(a) pyrene, Benz(a)-Anthracene and Quinoline.

PHYSICAL STATE: Suspended or dissolved in groundwater in low to trace quantitites.

HEALTH HAZARD INFORMATION: Although phenols are generally skin irritants which can exert toxic effects upon absorption into the body and PAH's have been associated with the production or cancers, these contaminants are likely to be present in low to trace concentration and therefore should pose no direct hazard to the sampling team. However, gloves should be worn during sampling to preclude skin contact.

CHEMICAL/PHYSICAL PROPERTIES: NA

TOPOGRAPHICAL HAZARDS: None known at this time

OPERATIONAL HAZARDS: None known at this time

## PERSONAL PROTECTION/TRAINING REQUIREMENTS

RESPIRATORY PROTECTION REQUIREMENT: LEVEL D

SPECIFICATIONS: _____

MODIFICATIONS: _____

PROTECTIVE CLOTHING REQUIREMENT:

- [X] WORK CLOTHES/COVERALLS (long sleeved)
- [ ] CHEMICAL PROTECTIVE CLOTHING. TYPE? - _____
- [X] WORK SHOES (STEEL TOE/SHANK)
- [ ] BOOTS. TYPE? _____
- [X] GLOVES. TYPE? Nitrile for collection of contaminated samples
- [ ] HARD HAT
- [ ] FACE SHIELD
- [ ] SAFETY GLASSES/GOGGLES

MODIFICATIONS: _____

TRAINING REQUIREMENTS: No special health and safety training required.

## AIR MONITORING REQUIREMENTS

1) INSTRUMENT: NA

2) INSTRUMENT: NA

MONITORING PROCEDURE:

## DECONTAMINATION PROCEDURES

EQUIPMENT/SOLVENTS/SOLUTIONS: NA

DECONTAMINATION PROCEDURES(S):

1) ITEM(S): NA

PROCEDURE:

2) ITEM(S):

PROCEDURE:

DISPOSAL PROCEDURE: General refuse for all consumables

# EMERGENCY REFERENCE

AMBULANCE: 911

POLICE: 911

FIRE: 911

HOSPITAL: Methodist Hospital
Location: 6500 Excelsior Blvd.
St. Louis Park, Minnesota
932-5000

DIRECTIONS TO HOSPITAL: MAP INCLUDED? No
Will vary depending on location of well

POISON CONTROL CENTER: 347-3141

NATIONAL RESPONSE CENTER: 1-800-424-8802

---

o ERT REPRESENTATIVES:

| | | |
|---|---|---|
| | ERT/CONCORD, MA | 617-369-8910 |
| | -KEVIN POWERS (HSM) | X 314 |
| | | 617-773-0484 (Home) |
| | -SCOTT WHITTEMORE (QA) | X 291 |
| | | 603-888-1174 (Home) |
| | ERT/MINNEAPOLIS, MN | |
| | -WILLIAM GREGG | 612-541-1642 |
| | MPCA DOUGLAS J. ROBOHM | 612-296-7395 |

o AGENCY REPRESENTATIVE: EPA DANIEL J. BICKNELL 612-886-2511

o CLIENT REPRESENTATIVE: JAMES GRUBE 612-924-2511

NEAREST PHONE: Public phones in St. Louis Park

COMMUNITY RELATIONS PLAN

The initial sampling plan is to be completed in conjunction with the enactment of various other work tasks embodied in a proposed Consent Decree - Remedial Action Plan for Reilly Tar, Minnesota, N.P.L. Site. All community relations programs related to the performance of Consent Decree tasks shall be coordinated through the following individuals.

| | |
|---|---|
| United States | Ms. Judy Beck<br><br>United States Environmental Protection Agency<br>(312) 353-1325 |
| State of Minnesota | Ms. Susan Brustman<br><br>Minnesota Pollution Control Agency<br>(612) 296-7769 |
| City of St. Louis Park | Ms. Sharon Klumpp<br><br>City of St. Louis Park<br>(612) 924-2523 |
| Reilly Tar & Chemical Corp. | Mr. John C. Craun<br><br>Reilly Tar & Chemical Corp.<br>(317) 248-6426 |

# SECTION D

# COMMUNITY RELATIONS PLAN

# COMMUNITY RELATIONS PLAN

The initial sampling plan is to be completed in conjunction with the enactment of various other work tasks embodied in a proposed Consent Decree - Remedial Action Plan for Reilly Tar, Minnesota, N.P.L. Site. All community relations programs related to the enactment of the Consent Decree tasks shall be coordinated through the following agencies:

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| City of St. Louis Park | Ms. Sharon Klumpp<br>City of St. Louis Park<br>(612) 924-2523 |

6 The description of laboratory quality control is not adequate. It should be revised to address at least the following:

a) Initial calibration

b) Calibration check and frequency.

c) Required detection limit

Check vs QA Obj.

d) % Recovery of surrogate spike, matrix spike and matrix spike duplicate. — the acceptable control limits should be specified.

e) Precision of duplicate analysis

f) Preventive maintenance

g) Corrective actions.

1. The Project description is not sufficiently addressed. The following should be addressed.

a) The intended data usage.

b) Sampling rationale and design.

c) Sample matrix and parameters to be tested.

2. Project Organization and Responsibilities

a) The organization chart (Figure 1-1) does not include EPA, Region V.

b) The description of the project responsibilities is not adequate. It should be revised to include responsibilities for sample collection, field Quality control, overall QC oversight, data assessment, etc.

3. Quality Assurance Objectives

The acceptance limits for accuracy, precision, and completeness should be specified.

4 Sampling

a) Table 2-1 (Summary of sampling program)

The parameters specified in Table 4-7 (page 46 of 55) (the Expanded analyte list) are not included in the Data required column.

b) The sampling numbering system to be used is not included.

c) In Section 2.5.1, it indicates that the sample containers cleaning procedure used by ERT is described. Should a bottle blank be analyzed per set of samples to ensure that no contamination, if any, resulting from the incomplete bottle cleaning?

d) In Table 2-5, the control limits are not specified.

5 Laboratory Quality Control

a) On page 23 of 55, one bullet should be added for the matrix spike and matrix spike duplicate.

1. Need a summary of parameters to be test.

2. Intended data usage.

3. The data validation criteria is debatable.

4. Analytical methodologies not specified clearly.

5. We question how the sample is extracted. If 2 liter separatory funnel is used to extract 2 liter of water sample, there is no much room for organic solvent. What kind of reproducibility can you achieve?

6 matrix Spike is not specified.

**UNITED STATES ENVIRONMENTAL PROTECTION AGENCY**
**REGION V**

**DATE:** November 17, 1986

**SUBJECT:** Review of Quality Assurance Project Plan (QAPP) for the Reilly Tar and Chemical Corporation N.P.L. Site,St. Louis Park, Minnesota

**FROM:** James H. Adams, Jr., Chief
Quality Assurance Office

**TO:** Norman Niedergang, Chief
CERCLA Enforcement Section

ATTENTION: Dan Bicknell, RPM

This memo transmits our office's disapproval of the Quality Assurance Project Plan (QAPP) for the Reilly Tar and Chemical Corp. N.P.L. Site, St. Louis Park, Minnesota, which our office received on October 14, 1986. This QAPP is not acceptable because it contains several major deficiencies, which needs to be addressed. First of all, this QAPP is not written according to the EPA Guidance for QAPP preparation. This makes the review of this QAPP labourious because required information is scattered throughout the QAPP. Secondly, the goal of the project to be achieved is not clearly defined. The project objectives, the inteded data usages, the parameters to be tested, etc., are not addressed. We also have questions regarding the sample preparation and the criteria to be used for data validation. These are some of the deficiencies,which needs to be addressed. We suggested that a QAPP meeting should be arranged to resolved these deficiencies.

CC: M. Gade, WMD
T. Rutter, ERRB
S. Hong, CES

Sampling

1. Documentation
2. sampling procedure — not just QA/QC
3. # of samples — table of summary
4. Sampling numbering system
5. Sample handling — filtration, preservation
6. Shipment, packing
7. field measurement — include procedures utilized and QC

Analytical procedures

1. Chain-of-custody and laboratory security
2. Documentation of all analytical methodologies to be used. EPA method to be used can be referenced specified by Referencing provided the test procedure are to be followed exactly. Any modifications should be documented. Any non EPA methods to be used should be attached.
3. Laboratory QC
   a) calibration and frequency
   b) preventative maintenance
   c) Required detection limit.
   d) % recovery of surrogate spike, matrix spike and MSD
   e) preventative maintenance
   f) Corrective action
4. Sample preparation

Chain of Custody
- final evidence file

Data reduction, validation and reporting

Our Office will approve this QAPP when the following are addressed.

I. Project Description.

1. Describe the actual difference of end data usage from the consent decree. It is stated that the end data usage will be slightly different from the Consent decrees, but it did not specify what is the difference. (page 7 of 125).

2. Describe the criteria for the QA objectives. The objectives for each sampling at each wells are discussed; however, there is no criteria specified for QA objectives.

II Project Organization and Responsibility

Identify the responsible parties for the Laboratory QC coordinator, field sampling personnel (page 10 of 125).

III. Sampling procedures (Section 6)